In-Situ Mechanical Property Evaluation of Dielectric Ceramics in Multilayer Capacitors

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ABSTRACT

The Young's modulus, hardness, and fracture toughness of barium titanate dielectric ceramics in three commercially available multilayer capacitors (MLCs) were measured in-situ using indentation and a mechanical properties microprobe. The three MLCs were equivalent in size (0805), capacitance (0.1 μ F) and dielectric type (X7R). The Young's modulus and hardness of the dielectric ceramics in the three MLCs were similar, while there were statistically significant differences in their fracture toughnesses. The results provide insight into the assessment of MLC mechanical reliability, and show that equivalent electrical MLC rating is not necessarily a guarantee that the dielectric ceramics in them will exhibit equivalent mechanical performance.

INTRODUCTION

Ceramic multilayer capacitors are complicated structures consisting of several different materials. If the mechanical reliability of the ceramic constituents in the MLC limits its electric service function or reliability or its manufacturability, then there is merit in understanding and predicting their mechanical performance. Understanding the mechanical reliability of ceramic MLCs is complex, however, because many parameters can affect it. All these parameters may be grouped into two categories: material parameters that describe the dielectric ceramic's mechanical properties and design parameters that can dictate what (thermo)mechanical stresses are imposed on the MLC during its service or manufacture [1-2]. The present study focused on parameters in the former category.

The mechanical performance of dielectric ceramics in MLCs is long recognized as being important to dependable service life; however their evaluation has become experimentally more complicated over the last

several years. Freiman and Pohanka [3] published an excellent review approximately 10 years ago on the mechanical properties of barium titanate (BaTiO_3) ceramics.

The further-miniaturization of MLCs since Freiman and Pohanka's review has resulted in (1) new MLCs and dielectric ceramics that are much different from those investigated and (2) test techniques that need refinement for testing them. The grain sizes of BaTiO_3 dielectric ceramics have been greatly reduced, and that has allowed the dielectric layer thicknesses to be made significantly smaller as a consequence. This in turn has greatly increased the capacitance/volume ratio of contemporary MLCs. Due to the lessening size of dielectric ceramic layers, margins, and cover layers in contemporary MLCs, standard mechanical properties tests such as flexure strength, microhardness, and fracture toughness by indentation cannot be readily performed. Consequently, valid mechanical property measurement must be conducted on a much smaller scale.

Depending on their capacitance and operating voltage, MLCs can have a wide variety of physical sizes as shown in Figs. 1-2. The MLCs examined in the present study are the smaller of the two sizes shown in Fig. 1, and have an operation rating of 10V. The procedures and analyses are easily extendable to the larger-sized MLCs, shown in Fig. 2, which are candidates for automotive power electronics and operate at 500-600V.

PROCEDURES

The BaTiO_3 dielectric ceramics in equivalent MLCs (X7R, 0805, 0.1 μ F) manufactured by three different suppliers were mechanically characterized. Each MLC's manufacturer was kept anonymous, and designated in the present study as MLC-A, MLC-B, and MLC-C.

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Figure 1. Examples of 0805 and 1206 multilayer capacitors. The dielectric ceramics in 0805 0.1 μ F X7R capacitors were examined in the present study.

Figure 2. Examples of high-voltage, high-capacitance ceramic multilayer capacitors that are amenable for use in automotive power electronics.

MLC SAMPLE PREPARATION

The MLCs examined possessed an orthotropic symmetry, so several (from each of the three manufacturers) were sectioned in half along each of the three orthogonal planes to completely reveal their internal electrode/dielectric geometry (shown in Fig. 3). A fine surface finish was a prerequisite for the planned micromechanical testing. To achieve this, the MLCs were sectioned, then set in metallographer's mounting epoxy and diamond-paste polished to a 0.25 μ m finish.

IN-SITU MECHANICAL PROPERTY TESTING

A mechanical properties microprobe (MPM)² was used in the measurement of the Young's modulus (E), hardness (H), and for the determination of fracture toughness (K_{Ic}) of the dielectric ceramic in each of the three MLCs. The MPM is an automated instrument that consists of four primary components shown in Fig. 4.: an indenter whose vertical displacement (nanometer resolution) and applied load (microgram resolution) are controllable, an optical microscope (OM) with several objective lenses, a precision X & Y stage that translates the metallographically prepared specimen between the OM and the indenter, and a computer that controls the OM's lens turret (50-1500x magnification), stage movement, and indenter load and displacement. A diamond Berkovich indenter was used for the E and H measurements, while a diamond Vickers indenter was used for the K_{Ic} measurements. The computer also collects data on the indenter's displacement and load, and is also interfaced with a TV-monitor and camera that show the microscope's field of view and allow inspection of each indent.

The MPM (not including the computer) is housed inside an insulated cabinet that minimizes its susceptibility to laboratory room temperature fluctuations and vibrations (problematic when controlling displacements at the nanometer level). The computer is outside the insulated cabinet to further minimize the introduction of thermal instabilities, and remotely operates the MPM.

Figure 3. The multilayer capacitors were orthogonally sectioned to reveal their internal layout.

² Nanoindenter II, Nano Instruments, Oak Ridge, TN

Figure 4. A mechanical properties microprobe was used for the testing.

A load-displacement curve is obtained for each indent. The relative contribution of elastic and plastic deformation can be assessed from this curve, and values for H and E determined.

For the present testing, a cover layer in each MLC was chosen to be subjected to indentation. Crack growth during fracture toughness testing can be affected by residual stresses; consequently, the cover layer region was chosen to be at a location (*i.e.*, as far away from the termination metal as allowed) where their effects were at a minimum.

Additionally, the plastic deformation zone created in the volume of material immediately under the indent has a finite size. The cover layer region had a relatively large volume and each indent's plastic zone in this region was not restricted by edges; this provided another motivation to apply indents in this location. The dielectric layers between electrodes were quite thin, so indentation was not performed in that region owing to concerns that edge effects would result in misleading values of E , H , and K_{Ic} .

Young's Modulus and Hardness

Relatively low loads applied with the Berkovich indenter were used for the E and H determinations (no cracks produced), while higher loads were used for the K_{Ic} determinations (cracks purposely produced). The load/displacement history during indentation provided data which were interpreted with the aid of an appropriate model to calculate E and H of the material [4]. Forty indents were applied in the dielectric ceramic in each MLC's cover layer, and the corresponding average E and H were calculated for each of them. A typical indent array is shown in Fig. 5.

Fracture Toughness

Higher loads were applied with the Vickers indenter to produce cracks which emanated from the corners of the indent, and these cracks were used to determine K_{Ic} . The polished metallographical mounts were carbon-coated to improve conductivity of the sample. The indent-generated cracks were imaged at magnifications of 7000 and 10000x with a scanning electron microscope (SEM) to measure their lengths. An image of an indent and its generated cracks are shown in Fig. 6. The crack length, its corresponding applied indent load, and the measured E and H for each MLC dielectric ceramic were needed to calculate K_{Ic} using [5]

$$K_{Ic} = \zeta \left[\frac{E}{H} \right]^{\frac{1}{2}} \left[\frac{P}{c^{\frac{3}{2}}} \right] \quad (1)$$

where ζ is a material independent constant for the Vickers indenter, E is the elastic modulus, H is the hardness, P is the applied load, and c is crack length (see Fig. 6). Analyses performed by the authors showed that the use of Eq. 1 is valid for use with the small indents produced by the MPM [6]. The values of E and H used in Eq. 1 were those measured with the MPM-generated crack-free indents. Twenty indents were applied at high loads to produce cracks, and up to four cracks were produced per indent. However, many indents did not have four cracks, so the number of fracture toughness values comprising the average was less than eighty for all three sets.

The analysis of these properties using a Vickers indenter with a conventional microhardness tester is standard; however, such indents would be much larger than the sizes of the cover layers, margins, and end margins in these MLCs. The indents generated with a MPM on the other hand are much smaller than those generated with a standard hardness tester (a few microns versus hundreds of microns); therefore, the MPM is a more suitable probe for dielectric ceramic mechanical testing. The investigators of this study are not aware (*i.e.*, no literature references found) of the MPM being used by others to mechanically characterize the various dielectric constituents in a MLC, so the described test technique in combination with the analyses in this report may be the first of its kind.

Figure 5. Indents were applied to the cover layer region within the multilayer capacitors.

Figure 6. An enlargement of the boxed-region in Fig. 5. The lengths of cracks produced by the high-load indents were measured and used in the fracture toughness calculation.

RESULTS AND DISCUSSION

YOUNG'S MODULUS AND HARDNESS

The Young's modulus of the dielectric ceramic in the MLC-C was slightly higher (203 GPa) than that for the dielectric ceramics in the MLC-A (193 GPa) and MLC-B (192 MPa). The difference between the former and the latter two E's is statistically significant though small. The hardnesses of the dielectric ceramics in each of the three MLC systems were statistically equivalent. The average E and H for the dielectrics in the three investigated MLCs are listed in Table 1, along with their standard deviations.

Table 1. Measured mechanical properties of dielectric ceramics.

Property	MLC-A	MLC-B	MLC-C
Young's Modulus, E, (GPa)	193	192	203
-Average-	40 tests	40 tests	40 tests
Young's Modulus, E, (GPa)	8.5	9.2	7.7
-Stan. Dev.-	40 tests	40 tests	40 tests
Hardness, H, (GPa)	11.3	11.3	11.9
-Average-	40 tests	40 tests	40 tests
Hardness, H, (GPa)	1.0	0.9	0.6
-Stan. Dev.-	40 tests	40 tests	40 tests
Fracture Toughness, K_{IC} , (MPa $\sqrt{m}$)	1.36	1.11	1.53
-Average-	49 tests	65 tests	65 tests
Fracture Toughness K_{IC} , (MPa $\sqrt{m}$)	0.29	0.28	0.47
-Stan. Dev.-	49 tests	65 tests	65 tests

The Young's moduli of the dielectric ceramic and termination metal in each of the three MLCs are not direct indicators of the potential mechanical integrity of the MLCs. However, their values can provide some insights. Principally, if the dielectric ceramic in a MLC has a relatively large-valued Young's modulus, then the imposed stresses in it will be higher than if its E were lower-valued (for the same applied strain). The E of the dielectric in the MLC-C is approximately 5% larger than the E of the dielectrics in the MLC-A and MLC-B, so stress in the MLC-C would be approximately 5% more if the three MLCs were subjected to the same amount of strain (e.g., all three mounted MLCs flexed the same amount on an epoxy board). The differences in stress due to the differences in the dielectric ceramic E's of the three MLCs are probably not significant and not an indicator of predicted mechanical integrity among these three MLCs. The E's of the termination metal provide some insight into their ductile nature; often a lower-valued E for a metal is an indicator that it can deform more readily when subjected to stress. Furthermore, if a termination metal is more ductile, then it can more readily relieve stress concentrations. Utilizing termination metals in the MLC with greater ductility is preferable because minimizing tensile stresses in MLCs will lessen the likelihood of mechanical failure of the dielectric ceramics within them.

The hardness of the dielectric ceramics is also not a direct indicator of the potential MLC mechanical robustness, but its value can also provide some insight into the effects of porosity and grain size on it. Hardness is typically inverse to the amount of porosity. This is often observed and exploited with conventional microhardness testing where the indent samples a very large number of pores and is hundreds of microns in size. However, with indents in the present study only being a few microns in size (see Fig. 6), and only perhaps an order of magnitude larger in size than the pores, the relationship between hardness and porosity at the operating-size-scale of the MPM cannot be readily interpreted. Lastly, the measured hardnesses were more representative of the hardness of the BaTiO_3 grains because the MPM produced an indent size that sampled regions consisting of sub-micron grains; conversely, this is not always the case for microhardness testing because the large indent size it produces samples many more grains, but also porosity, grain boundaries, etc.

Qualitatively, if the average pore size is small, then the fracture toughness of the ceramic may be relatively high because crack growth may be inhibited or arrested by the pores. Conversely, if the average pore size is relatively large, then the pores may act as actual strength-limiting flaws with the effect of lowering the strength of the ceramic. The hardness of the dielectric ceramics in the three examined MLCs were in fact equivalent, so if these were active effects, then they were equivalent in the three different dielectric ceramics.

FRACTURE TOUGHNESS

The K_{Ic} of the MLC-C (1.53 $\text{MPa}\sqrt{\text{m}}$) was larger than that for the MLC-A (1.36 $\text{MPa}\sqrt{\text{m}}$) which in turn was larger than that for MLC-B (1.11 $\text{MPa}\sqrt{\text{m}}$). The fracture toughness of another BaTiO_3 (as measured with microhardness indentation testing) was measured to range from 1 to 1.8 $\text{MPa}\sqrt{\text{m}}$ [7-8]. Fracture toughness in BaTiO_3 can depend on several variables including grain size, chemical composition, and crystal structure [3]. The differences among the average fracture toughnesses were statistically significant and conclusive. The average K_{Ic} values for the dielectric ceramics in these three MLC systems are listed in Table 1 and illustrated in Fig. 7. Lastly, brittleness and hardness have been shown to be interrelated [9] in that hard materials are more apt to be brittle (i.e., have lower fracture toughness); however, the statistically significant difference in fracture toughnesses in these materials did not manifest itself in different hardnesses, so this relationship is not evident in these barium titanate ceramics.

Ceramics with maximum K_{Ic} are preferable for load-bearing applications. Dielectric ceramics in MLCs may be subjected to tensile stresses during manufacturing or service, so using a dielectric ceramic with maximum K_{Ic} will minimize the likelihood of mechanical failure. A design window is schematically shown in Fig. 8 and it

illustrates that a larger-valued fracture toughness increases the design window or the device survivability. The dielectric ceramic in the MLC-C had the highest K_{Ic} , while the dielectric ceramic in the MLC-A had the second highest K_{Ic} and that in the MLC-B had the lowest. All other things being equal, the dielectric ceramic in the MLC-C would have the best mechanical integrity and would have the lowest likelihood of mechanical failure of the three MLCs if they were subjected to equivalent mechanical loadings.

CLOSING COMMENTS

Other factors, although not quantified in this study, can also affect mechanical performance and should be considered in the optimization of MLC mechanical robustness. Parameters such as electrode centering, cover layer and margin thicknesses, deformation

Figure 7. Fracture toughness comparison of the dielectric ceramic toughness in the three MLCs.

Figure 8. Fracture toughness comparison of the three MLCs.

behavior of the termination and electrode metals, residual stress, stress-free temperature, adhesion strength of adjoining laminates in MLC cover layers, thickness of laminates comprising the cover layer, glass frit effects, and thermal shock susceptibility can all affect MLC mechanical robustness [1-2, 10].

The authors believe that appropriate design of a MLC, in which service- or manufacturing-induced tensile stresses are minimized, will have more of an impact on improved MLC mechanical robustness than any improvement in mechanical properties of BaTiO_3 ceramics. Barium titanate ceramics were never intended to be a structural ceramic, so a drastic improvement of their mechanical properties is unlikely. The fracture toughnesses and strengths of structural ceramics, like those for silicon nitride or partially-stabilized zirconia, have doubled or even tripled over the years as their engineering has matured. However, it is doubtful that BaTiO_3 will enjoy the same fracture toughness and strength development because there is not an economic driving force to fund that (unlike for silicon nitride and zirconia). Appropriate or improved design to limit service tensile stresses will likely have more of an effect in the improvement of MLC mechanical robustness than tinkering with BaTiO_3 to make it twice as tough or strong. Engineering advances have increased the fracture toughness of structural ceramics up to 7-12 MPa $\sqrt{\text{m}}$; doubling the fracture toughness of BaTiO_3 would increase it only to 2.5-3 MPa $\sqrt{\text{m}}$. Additionally, structural ceramics can have flexure strengths larger than 1000 MPa, while the strengths of the strongest BaTiO_3 ceramics are an order of magnitude less [1]. Doubling the strength and fracture toughness of BaTiO_3 ceramics would be helpful; however, designing MLCs so that imposed tensile stresses are halved would result in the same mechanical robustness state, and would be much less of an engineering endeavor.

CONCLUSIONS

The present study consisted of the measurement of several mechanical properties of dielectric ceramics in MLCs. If MLC mechanical reliability is a limiting factor to long-term electronic function, then it is clearly advantageous to use a MLC with maximum mechanical robustness. Maximum fracture toughness is described here as an indicator of the robustness.

The Young's modulus, hardness, and fracture toughness of the dielectric ceramics in three equivalent (X7R 0805 0.1 μF) MLCs were measured in-situ. From a perspective of a structural ceramic designer, the authors conclude that if the three MLC sets were to be subjected to an equal service stress state that the dielectric ceramic in the MLC-C would mechanically perform the best, followed in turn by the dielectric ceramic in the MLC-A and then that in the MLC-B (all other things being equal). This conclusion is based on the dielectric ceramic in MLC-C having the highest fracture toughness.

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