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Nanomechanical characterisation of pervoskite nanocubes of $Na_{0.5}K_{0.5}NbO_3$ for fatigue analysis

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Abstract

In this present work, pervoskite nanocubes of $Na_{0.5}K_{0.5}NbO_3$ ceramic have been synthesized by new method were investigated by nanoindentation with a Berkovich indenter. The development of lead free ceramics has been one of the most important issue to current researchers. Among the studied lead free ceramics, sodium potassium niobate $Na_{0.5}K_{0.5}NbO_3$, alkali niobate based ceramic has been found to be the most promising candidate because of its piezoelectric, as well as good electrical properties. Much attention should be focused to the study of surface nanomechanical properties of such materials, because the success of many applications is partly determined by a precise understanding of these characteristics. Therefore, determination of the surface and mechanical properties of $Na_{0.5}K_{0.5}NbO_3$ ceramic material becomes more important. Among conventional methods used for mechanical characterization, such as the bulge test and microbeam techniques, nanoindentation methods have gained widespread use because of the ease of sample preparation and the inherently simple estimation of hardness, stiffness, elasticity, etc. Nanoindentation has been established as a quantitative tool for the characterization of mechanical properties of materials on the nanoscale. Therefore, the nanoindentation test has been carried out to measure displacement produced in the sample in response to the force applied at different locations on the $Na_{0.5}K_{0.5}NbO_3$ surface using nanoindentation set up. We have conducted nanoindentation based fatigue analysis on $Na_{0.5}K_{0.5}NbO_3$ Pelletes sintered at 1000°C for multiple cycles of load and the results are discussed in the manuscript.

Introduction

The analysis method proposed by Oliver and Pharr in early 1990's has been considered as one of the standard method for determining the elastic modulus and hardness from the indentation load-displacement curves for materials[1-3]. Requirement of nanoindentation arise from the necessity to measure the surface and mechanical properties of ultra thin films as well as very small volumes of materials[4]. The nanoindentation technique have been widely used to understand surface nature and mechanical properties at nano scale as well as micro scale thin films as well as elastic and plastic properties of ceramic pellet material.

Elastic modulus (E), and hardness (H) are the two major mechanical properties are generally determined using nanoindentation techniques and there has been considerable progress in the measurement of other mechanical parameters as well, including hardening exponents, creep parameters, relaxation, yield strength, stiffness and residual stresses[5-7]. Fracture toughness can also be determined using nano indentation technique by fracture mechanics approach, similar to that applied to ceramics and semi-brittle materials[8]. Even though other technique such as AFM nanoindentation is available, they are not so widespread for the study of mechanical properties of materials at the nanoscale. In the last two decades, however, a veritable revolution has occurred in indentation testing, owing to the development of new sensors and actuators that allow instrumented indentations to be routinely performed on nano scales. The resulting technique, termed nanoindentation, has now become ubiquitous for mechanical property measurements at surface of various materials including ceramics, polymers, semiconductors, MEMS, etc.

The major components in a nanoindentation experiment are the sample, the sensors and actuators used to apply and measure the mechanical force and indenter displacement, and the indenter tip. The indenter tip is conventionally made of diamond, formed into a sharp, symmetric shape such as the three-sided Berkovich pyramid. Nanoindentation is a diversity of indentation rigidity tests applied to very small volumes. The indentation is one of the common method used to test the mechanical properties of ceramic. Hence we carried out the nanoindentation studies on pervoskite nanocubes of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellets.

Experimental

For the nanoindentation studies, we weigh about 0.5 gram of $Na_{0.5}K_{0.5}NbO_3$ nanomaterial. The powders are made into pellet having diameter of about 10mm, and thickness of about 2mm using pellet making machine. To attain necessary mechanical strength for $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet, we have sintered it up to 1000˚C for 3 hours. The snap shot of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet loaded on the stage during nanoindentation experiment is shown in figure 1.

A berkovich nanoindenter tip which has a three-sided pyramid geometry is used for testing the indentation hardness of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet material. We used Oliver and Pharr method (power law method) for the nanoindentation of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet. The nanoindentation experiment is carried out on the CMS instruments UNHT (Anton Paar). The

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complete view of UNHT and microscope on CSM instrument load with $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet material is shown in figure 2.

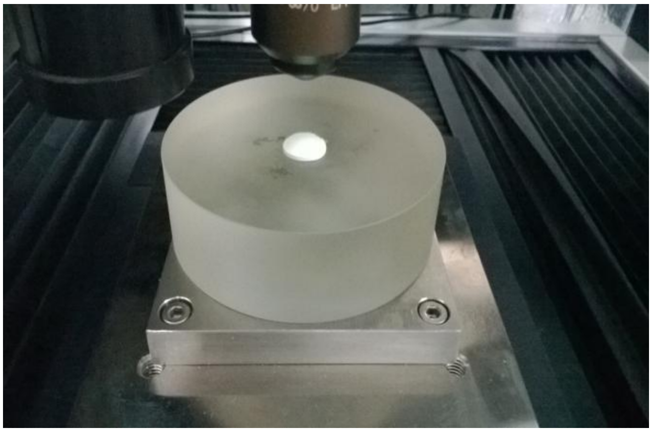

Figure 1: The snap shot of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet during experiment

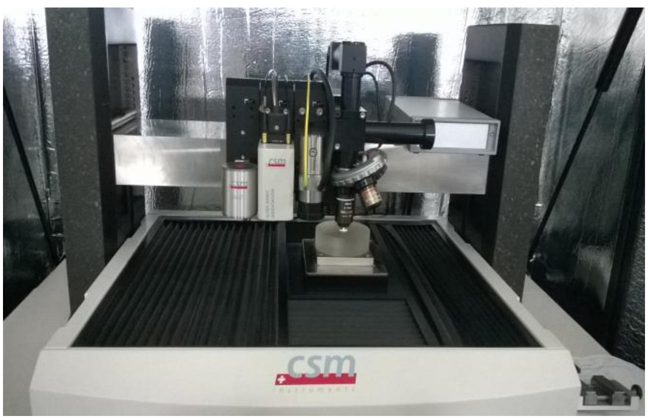

Figure 2: The snap shot of Micro Hardness Tester CSM Instruments UNHT loaded with $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet

We have performed two types of experiments, such as (1) Single cycle loading-unloading and (2) Multiple cycle loading-unloading. For single cycle loading-unloading experiments, we have used maximum force of 5mN with loading rate of 10 mN/min, unloading rate of 10 mN/min, and pause of 2 seconds between loading and unloading cycle. When the system senses a force of 0.1 mN on the ceramic pellet sample, it will start acquiring the applied force vs penetration depth at an acquisition speed of 10 Hz. For multiple cycle experiment, we have applied maximum load of 5 mN for loading and pause for 10 seconds. After the pause for 10 seconds, we have unloaded the force to 2 mN and then increased to 5 mN. This loading and unloading process is repeated for 30 cycles. Similar to single cycle experiment, when the system senses a force of 0.1 mN on the ceramic pellet sample, it will start acquiring the applied force vs penetration depth at an acquisition speed of 10 Hz. During multiple cycle experiments also we have used loading rate of 10 mN/min during loading curve and unloading rate of 10 mN/min during unloading curve.

Results and Discussion

$Na_{0.5}K_{0.5}NbO_3$ ceramic pellet having good surface uniformity is focused with the help of optical microscope attached to CSM instrument and we choose the locations for indentation on the ceramic pellet. After the indentation on ceramic pellet with a maximum force of 5mN, berkovich mark is observed on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet, which is shown in figure 3.

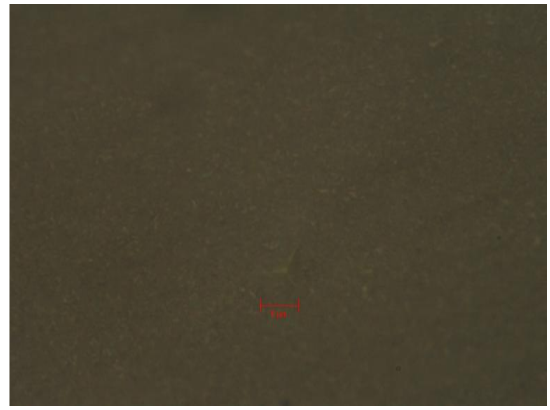

Figure 3: The snap shot of corresponding microscopicy image of the Berkovich indent with cracks.5 micro meter, (maximum load= 5mN)

Hardness Analysis

Hardness tests are widely used in field of mechanical and surface engineering to determine hardness properties of ceramic materials. Hardness is a measure of how resistant solid matter is to various kinds of permanent shape change when a compressive force is applied.

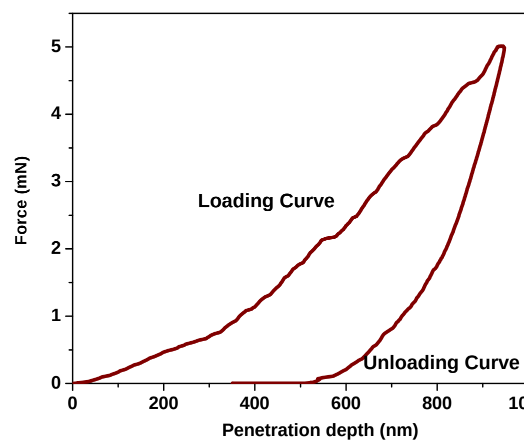

Figure 4: Force (mN) versus penetration depth (nm) observed during nanoindantation of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet

Macroscopic hardness is generally characterized by strong intermolecular bonds, but the behavior of solid materials under force is complex; therefore, there are different measurements of hardness: scratch hardness, indentation hardness, and rebound hardness. During a typical nanoindentation test, force (mN) vs penetration depth or displacement (nm) are recorded as the indenter tip is pressed into the ceramic (i.e. Pellet) material's surface with a prescribed loading curve and unloading curve profile.

While applying 5 mN load on ceramic pellet it penetrates up to 949 nm during loading and after unloading a permanent deformation of around 523nm is observed. Force (mN) versus penetration depth (nm) observed during nanoindantation of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet is depicted in Fig. 4. The applied force vs time is shown in figure 5. The penetration depth (nm) versus Time (sec) on $Na_{0.5}K_{0.5}NbO_3$ ceramic is shown in figure 6. The observed parameters from the loading and unloading curve is tabulated in table 1.

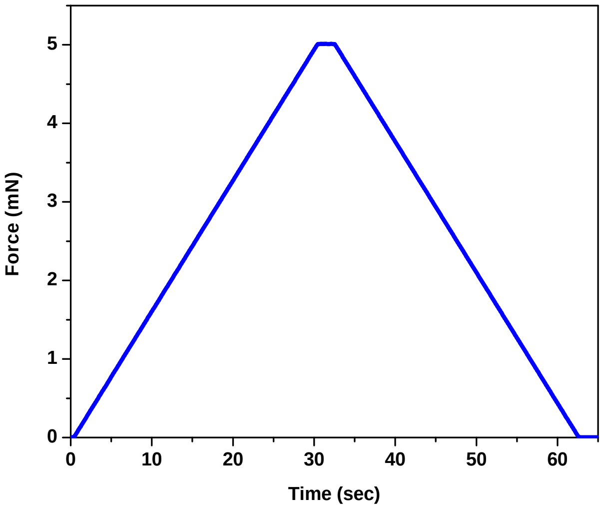

Figure 5: Applied force (mN) versus time (sec) on $Na_{0.5}K_{0.5}NbO_3$ ceramic

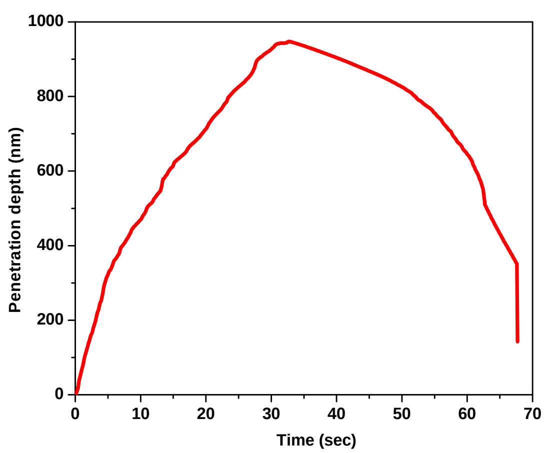

Figure 6: Penetration depth (nm) versus Time (sec) on $Na_{0.5}K_{0.5}NbO_3$ ceramic with maximum force of 5mN

Table 1: Observed parameters from the loading and unloding curves of nanoindentation on $Na_{0.5}K_{0.5}NbO_3$ ceramic

Properties	Result
Indentation Hardness (HIT)	365.868 MPa
Indentation Elasticity (EIT)	5.734 GPa
Indentation Creep (CIT)	1.137 %
Contact Stiffness (S)	0.029 mN/nm
Elastic work done (W_{elast})	633.525 pJ
Plastic work done (W_{plast})	1182.536 pJ
Maximum penetration depth (h_{max})	949.7966 nm
Permenant indanation depth (h_p)	523 nm

Fatigue analysis

Figure 7. shows the influence of multiple cycle load and indentation response $Na_{0.5}K_{0.5}NbO_3$ ceramic. In this experiment we have used 30 cycles of 5mN – 2mN force. In between two successive cycles, we have made a pause of 10 seconds. During the pause of 10 seconds in the first cycle with applied force of 5mN, we have observed a sudden penetration causing creep on the sample is observed as shown in figure 7. Hence 5mN can be considered as a fatigue limit for this material. Also hardness and elasticity is less which can be concluded from figure 7. As the loading curve is uneven during the first cycle, confirms that compactness of sample is less uniform.

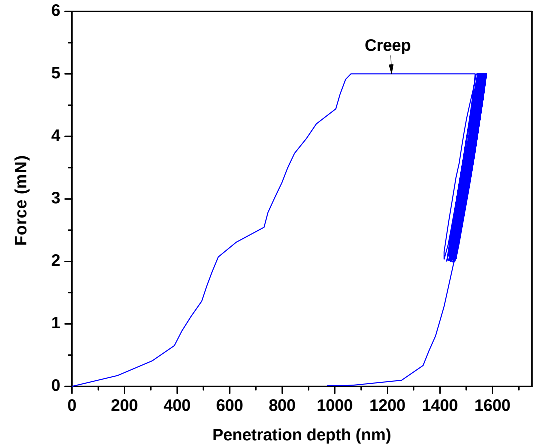

Figure 7: Schematic drawing of Force (mN) versus Penetration depth (nm) during nanoindentation of ceramic material such as $Na_{0.5}K_{0.5}NbO_3$ pellet

At the end of 30^{th} cycle of varying load, maximum penetration depth of 1571 nm is observed. After the complete unloading of applied force, permanent penetration depth of 1256 nm is observed on the sample. In the multiple cycle experiment, applied force vs time used on the sample is shown in figure 8. During the multiple cycle, the penetration depth (nm) versus Time (sec) on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet is shown in figure 9.

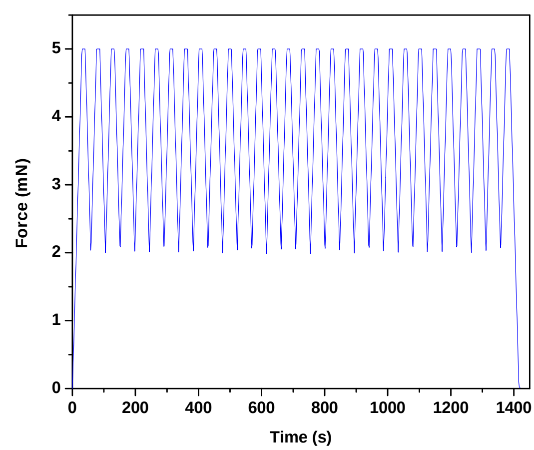

Figure 8: Force (mN) versus Time (s) used during multiple cycle experiment on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellets

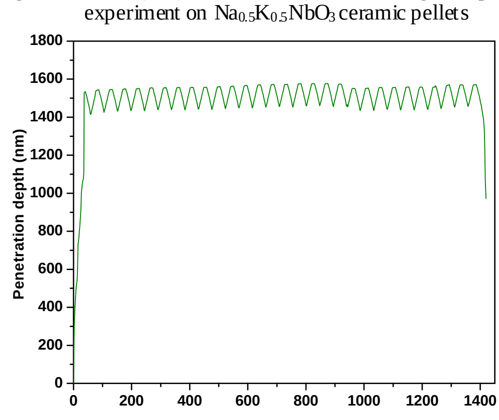

Figure 9: Penetration depth (nm) versus Time (s) observed during multiple cycle experiment on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellets

The hardness of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet specimen, plotted against penetration depth or depth of contact is shown in figure 10. It clearly shows that the hardness decreased from 117.8 MPa to 111.83 MPa at the end of 30 cycles. On fitting the data points on linear fit, we got the equation y= -0.1653x + 351.99 with a regression coefficient r^2=0.99983, which shows the decrease is linear.

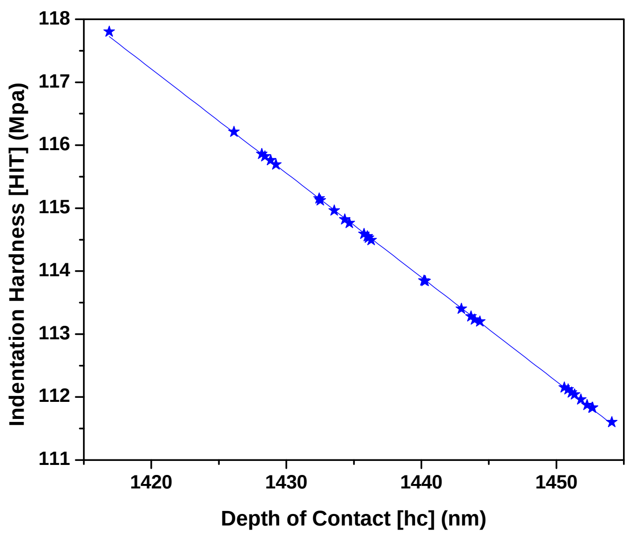

Figure 10: Indentation hardness (MPa) versus Depth of contact (nm) during multiple cycle loading-unloading on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellets.

The indentation modulus (EIT) during multiple cycle of loading is not drastically varying and it is shown in figure 11. Permanent indentation depth (hp) after removal of test force during multiple cycle of loading on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellets is also not significantly changing as shown in figure 12. Hardness at the end of each cycle is computed and plotted in figure 13. From the figure 13 it is observed that the hardness initially decreases upto 17 cycle, afterwards the hardness is increasing upto 21 cycle, then again it decreases.

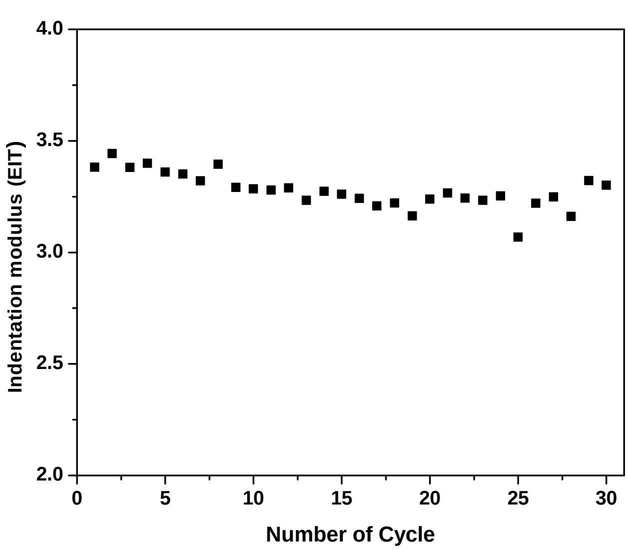

Figure 11: Variation of Indentation modulus during multiple cycle of loading

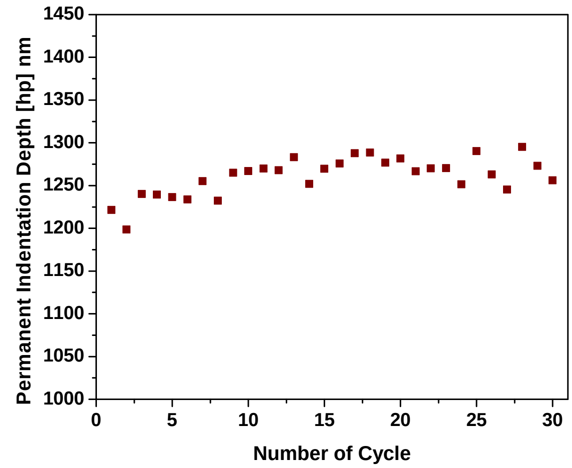

Figure 12: Permanent indentation depth [hp] (nm) versus Number of Cycle

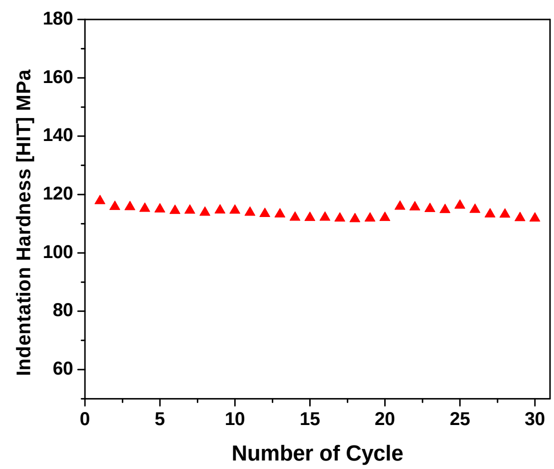

Figure 13: The plotted graph is shows that HIT versus Number of Cycle

Conclusions

Indentation fatigue on $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet with a berkovich nanoindenter tip is investigated by using Oliver and Pharr method. The pervoskite nanocubes of $Na_{0.5}K_{0.5}NbO_3$ used for present experiment is synthesized at our lab. Force vs penetration depth based indentation instruments provide a means for studying the plastic, elastic and fatigue properties of $Na_{0.5}K_{0.5}NbO_3$ ceramic pellet. We have performed two types of experiments (1) Single cycle loading-unloading and (2) Multiple cycle loading-unloading. The results of both the type of experiments shows the various nanomechanical characteristics of $Na_{0.5}K_{0.5}NbO_3$ such as creep, hardness, elasticity, stiffness, indentation modulus.

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