공학석사 학위논문

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결함 분포에 관한 연구

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이 논문을 공학석사학위논문으로 제출함

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Abstract

A study of defect distribution of $BaTiO_3$ dielectrics in MLCC by TEM-CL

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Cathodoluminescence (CL) is an electron stimulated emission of photons ranging from x-rays to nearly infrared rays from materials that are solid in nature. In this regard therefore, the radiation generated from ultraviolet to mid-infrared in the electromagnetic spectrum is known as CL. From the resultant luminescence, either spatial or spectral analysis can be obtained. It is one of the commonly applied advance techniques found to be highly flexible since it offers a useful analysis of variety of ceramic oxides,

alternatively referred to as perovskite glass ceramics such as $BaTiO_3$, $SrTiO_3$, $PbTiO_3$[8]. In addition, CL is used to study fundamental aspects of light transport, electronic structure of substances which may include semiconductors and scattering among other aspects. In this regard therefore, CL acts a helpful source of information which can be used for fundamental research and applied research which has a direct link to industry, for instance, metrology and failure analysis.

With growing of Multilayer Ceramic Capacitor (MLCC) market, the reliability of the product became a great issue of concern because more reliable data about defects was needed. A lot of studies about $BaTiO_3$ dielectrics in MLCC were done with cathodoluminescence technique but they were limited to Scanning Electron Microscopy-Cathodoluminescence (SEM-CL). Due to the limitation of SEM-CL regarding low magnification and low resolution and the fact that Transmission Electronic Microscopy (TEM) does not have such limitations, home-built TEM-CL was chosen to investigate BTO dielectrics. In this regard therefore, $BaTiO_3$ dielectrics in MLCC were investigated using TEM-CL.

The investigation revealed two aspects; firstly, the band gap structure and energy levels were revealed. Through monochromatic CL images of each types of defects (oxygen and barium vacancies), their distribution and relative concentration were visualized. In addition, two unreported peaks were found. The explanation for this could be probably because of the fact that TEM had better resolution and higher magnification as compared to SEM.

Regarding the two unreported peaks which were found, we estimated that they were from dopants since in MLCC, many elements are added for reliability. To identify the origin of the two unreported peaks, the monochromatic images were subjected to further investigations along with Energy Dispersive X-Ray Spectroscopy(EDS) mapping. The peaks were found to be Zirconium (Zr) and yttrium (Y). Their concentrations and distributions were also visualized.

It is thought that these results can benefit in visualization of invisible defects as well as understanding of the emission behavior of $BaTiO_3$ microstructure.

Keyword: Cathodoluminescence, TEM, $BaTiO_3$, defect distribution, MLCC, grain boundary

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Chapter 1. Introduction

1.1 Trends of multi layer ceramic capacitor

The modern society has much regard for materials that have small sizes and light weight and for portable electronics that can tackle a number of tasks simultaneously. Notably, one aspect that makes people currently to have a great aspiration for enhanced gadgets is due to efficiency. Electronic substances that can consume low amount of electric power and can have increased speed while tasking have critically become vital.

As such, one of the techniques applied in fulfilling these demands is by utilizing thin film multi-layering technology. Notwithstanding the demerits linked to application of multi-layer ceramics capacitors, it has been used effectively to enhance the portable computers, mobile phones and other electronic materials.

Multi-layer ceramic capacitors (MLCC) have hugely been applied in the mobile phone industries, portable computers, filter circuit fields, and coupling circuits. MLCC products that have utilized $BaTiO_3$ have widely been used in the field of direct current (DC) electric materials, for instance, Z5U and EIA-Y5 V[12].

Also, It is worth noting that design engineers would likely prefer ceramics such as $BaTiO_3$ and $SrTiO_3$ due to their stability and reduced costs. Moreover, within a circuit, ceramics tend to have an extended performance, hence making them to be a choice for many the contemporary technology engineers[14]. However, Miniaturization, which is one of the methods that have permitted use of electronics that have very little sizes, it still has its demerits since the dielectric substances that have decreased thickness have a high possibility of starting to leak.

While Ni Oxides and $BaTiO_3$ dielectrics are co-fired predominantly to escape oxidation, they create oxygen vacancies in the reducing atmosphere. Particularly, within MLCC, inner electric and electrostrictive telescopic stress takes place, making the dielectric to breakdown due to cracking that results from accumulation of oxygen vacancies to cathode. The problems that are associated with application of multilayer ceramic capacitors, encompasses current leakages and short circuiting. However, huge permittivity can be achieved which then leads to low dielectric loss and is notable within $BaTiO_3$ ceramics that are rare earth-doped[13].

To mitigate the cracking and breakdowns issues that are usually notable within multi-layer ceramic capacitors, many researches about dopants have been conducted to enhance the dielectric properties.

Wang reported that metal components Nb^{5+}, Mg^{2+} and Sr^{4+} can be used instead of the Ti^{4+} which leads to creation of oxygen vacancies. Consequently, the energy that is generated from the lattice change creates separation of the grain boundary, which at the end of the process ensures that the grain does not grow[12].

Choi reported that permittivity is a necessity for an efficient

MLCC and can be achieved via the incorporation of Ga^{3+} to increase it, since it forms prominence within the transition[1].

Jeon undertook an experiment undertaken by predominantly to highlight about that included the core-shell structure prepared in $BaTiO_3$ ceramics, so as to review the doping impact on both the structure and the dielectric properties within the $BaTiO_3$ ceramics[4].

Leto suggested that via cathodoluminescence, is it possible to realize the morphological changes that comprise the relative intensity of $BaTiO_3$, to highlight how it fluctuates in wet and dry temperatures. Full width at half maximum (FWHM) of the $BaTiO_3$ sub-bands are also likely to show morphological changes while induced in firing temperature[8].

It has been known that since cathodoluminescence spectrum has a suitable sensitivity to mechanical and chemical gradients, it is the most suitable method to study about the effects of multilayer ceramic capacitors[8].

1.2 Cathodoluminescence

Cathodoluminescence is an electron stimulated emission of from materials. A Cathodoluminescence (CL) technique plays a significant role in evaluation of any possible defect structures. From the resultant luminescence, either spatial or spectral analysis can be obtained. When it is equipped with Transmission Electron Microscopy (TEM), its ability to combine the functional optical information together with the superior spatial resolution enables the technique to effectively help in assessing the defect structures.

In this paper, $BaTiO_3$ dielectrics in MLCC was investigated by TEM-CL. Distribution of vacancy defects as well as band gap structure and transition of each types of defects were discussed. In addition, two unreported CL peaks were found and identified.

1.3 Experimental details

The commercial MLCC with X7R temperature characteristic by Murata electronics. was used. Sample was mechanically polished down to 100nm thickness. Then ion milling was done with Gatan Precision Ion Polishing System. Total milling time was 23 miniute and LN_2 was used for protection. JEM-2010F was used for TEM analysis. TEM-CL was conducted with our home-built TEM-CL holder with LN_2 dewer attached for cooling. Shamrock 163 spectrometer by Andor Solis was used for acquisition of CL signals and home-built software called QTCL was used to acquire spatial CL data. CL signal was acquired for 500 seconds and 200msec/pixel for spectrum and map at respectively. CL investigations were performed at 120kV and LN_2 was used for all CL experiments.

Figure 1. Home-built TEM-CL holder

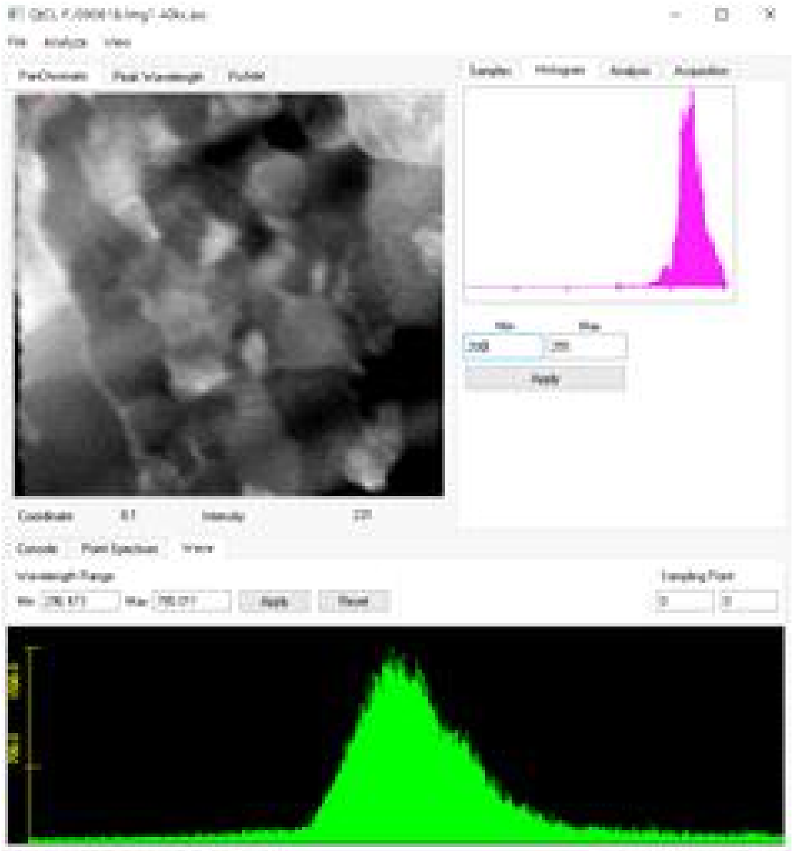

Figure 2. Home-built software,“QTCL”

Chapter 2. Idea of finding atomic-level defects

2.1 Cathodoluminescence spectrum analysis

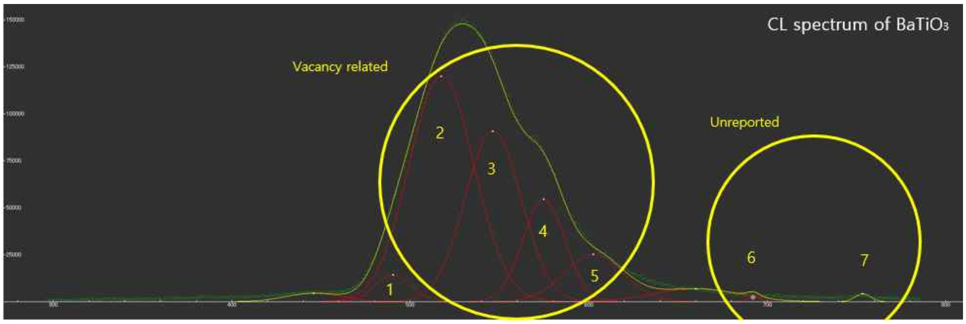

Figure 3. Cathodoluminescence spectrum of $BaTiO_3$

Figure 3. shows the acquired CL spectrum for $BaTiO_3$ dielectrics in the commercial MLCC. The spectrum is in a good agreement with generally known $BaTiO_3$ spectrum shape and already known transitions due to vacancy defects[9]. The peak before peak 1 is assumed to be originated from transition above band gap range. Peak 1 was caused by band gap transition. From peak 2 to 5, they

center at 517, 546, 575 and 602 respectively. Their transitions correspond to V_o^+ to V, C to V_{Ba}^-, V_o^+ to V_{Ba}^- and C to V_{Ba}^- respectively. Moreover, 2 unreported peaks were found namely peak 6 and peak 7[6]. This foundation is thought to be due to the better resolution of TEM than that of SEM and will be discussed in part II.

2.2 Cathodoluminescence image and distribution of $BaTiO_3$

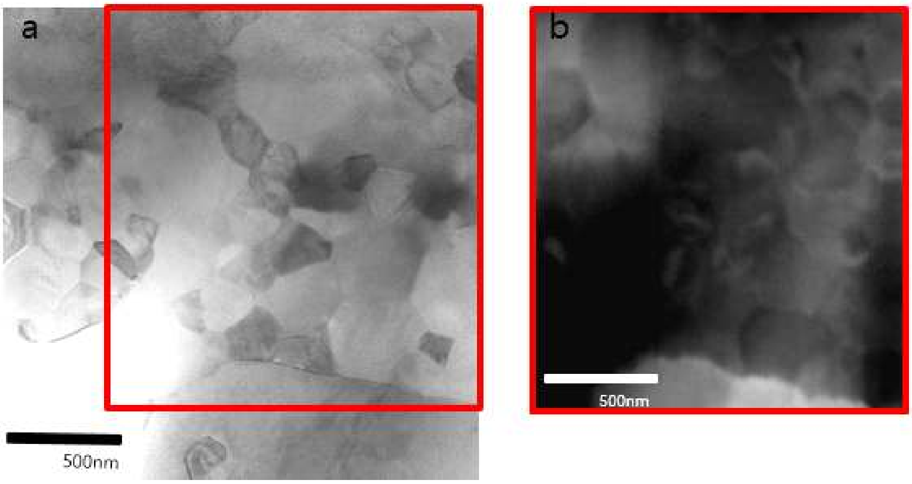

Figure 4. (a). STEM BF and (b). Panchromatic image of $BaTiO_3$

Figure 4. (a) and (b) are the STEM bright field image and corresponding panchromatic CL Image of boxed area respectively. Figure 4. (b) shows distribution and relative concentration of defects. Strong grain boundary emission is well shown and this is reasonable because, thermodynamically, defects tend to segregate in the grain boundary for their lower activation energy than grain interior. Also, not only emission on grain boundaries where defects are concentrated but emission on grain interior can be well observed. Above the nickel electrode, emission in grain boundary was not detectable. This is due to the different amounts of electron scattering with respect to different zones and emission on Ni electrode is due to the oxidation.

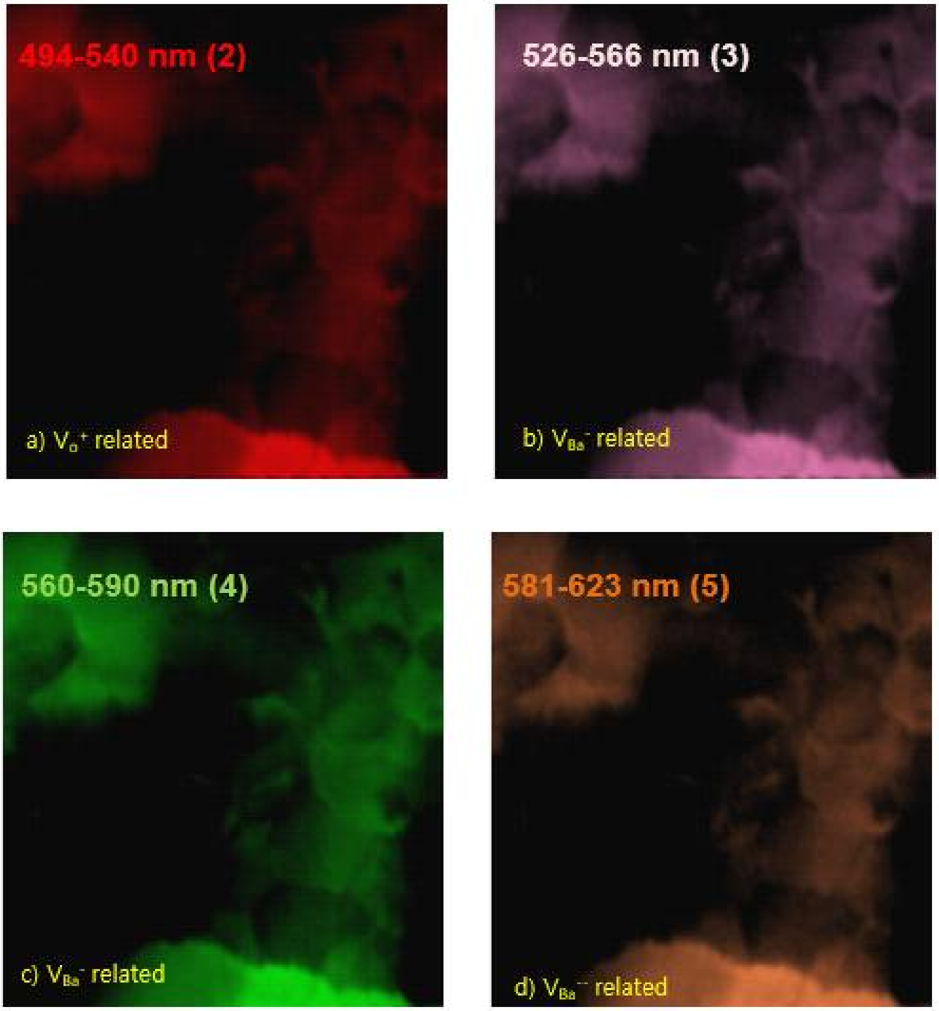

Figure 5. Monochromatic images of each types of vacancy defects.

Monochromatic images of each types of defects are also illustrated in figure 5. a-d. Monochromatic images show stronger emission in grain boundaries as well, meaning oxygen and barium vacancies are segregated along the grain boundaries. However, their positional differences were hard to tell.

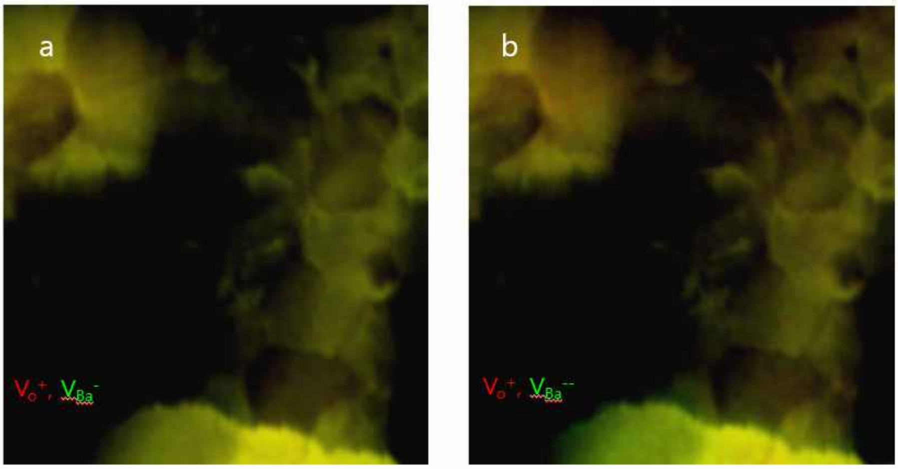

Figure 6. Distribution of each types of vacancy defects

The figure 6 is the composite image of oxygen and barium vacancies. For a and b, the colors of V_o^+ and V_{Ba}^- were switched for clear distinction. Position and distribution of oxygen and barium vacancies are clearly distinguished as reddish or greenish regions, meaning it is possible to figure out the location of invisible defects.

Also, yellowish regions indicate that oxygen and barium vacancies are so closely located that their emissions overlap.

2.3 Contour map

The contour map in figure 7 demonstrates the CL intensity along the yellow line in panchromatic image. Strong orange color is demonstrated among 515-545 nm wavelength, which is included in oxygen vacancy related wavelength(494-540nm). The spectrum intensity is related to the amount of each types of defects. As the amount of each defects increases, intensity increases and the amount of certain types of defects changes, spectrum shape is changed[3,10]. From this, it can be inferred that the presence of oxygen vacancies is dominating in the sample. As thus, virtually invisible vacancy in the microstructure was also confirmed by visualizing the relative concentration.

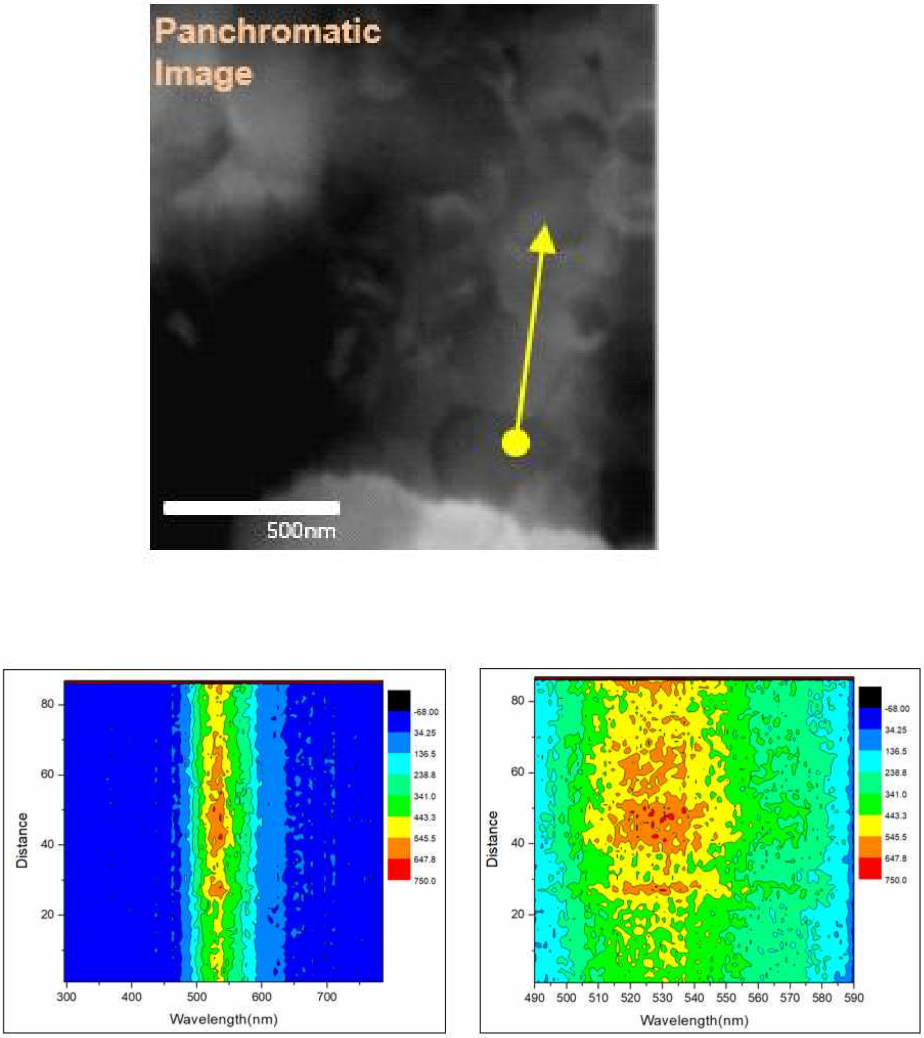

Figure 7. Panchromatic image with CL line scan region and corresponding contour map

Chapter 3. Identification of unreported peaks

3.1 Origin of unknown peaks

As mentioned in part I, 2 unreported peaks were observed. It is expected that due to the characteristics of TEM, which is high in magnification and resolution, those peaks were observed. Also it is expected that the peaks were originated from impurities such as dopants because a lot of elements are added in MLCC for reliability. The reason is that even though the presence of impurities does not imply that they are not necessarily contribute to the occurrence of new CL transition, new kind of energy levels can be introduced by impurity substitution[7].

The commercial $BaTiO_3$ is made in the form of $(Ba_{1-x}A_x)(Ti_{1-y}B_y)O_3$ for reliability. The A element can be substituted into Ba site and the B element can be substituted into Ti site for their purposes. Huang, in fact, reported that transition metal is mainly used as element B, which is reported to produce deep trap levels[2].

3.2 Monochromatic image and EDS mapping comparison

To find out the trend of elements, EDS line scan was done as shown in figure 8. Figure 8 illustrates that elements such as Ba, Y, Mg, Zr and Dy are detected, showing different height across the grains. It is because of different scattering effect as mentioned earlier. Dopants are also segregated along the grain boundaries that we focused our analysis on grain boundary emission.

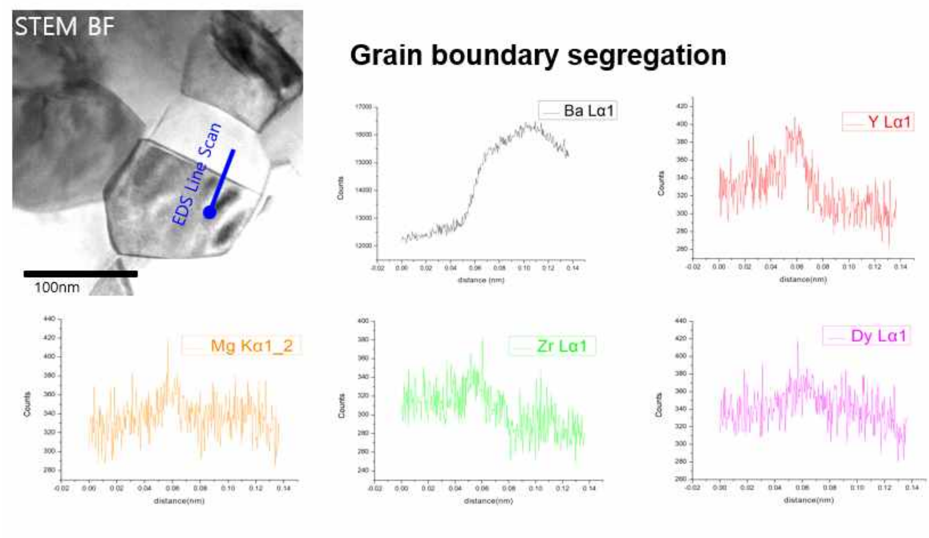

Figure 8. EDS line scan across the grain boundary

After we observed the grain boundary segregation of elements, we compared the monochromatic of different wavelengths around the grain boundary. In figure 9, the magnified image, compare with the magnified monochromatic image for 748-758 nm, a clearer emission characteristic is detected in the 688-696nm monochromatic image. In the CL image, the emission of a specific region should represent that origin of the emission is in the region. As thus, we conducted EDS mapping to confirm that the emission was caused by the element.

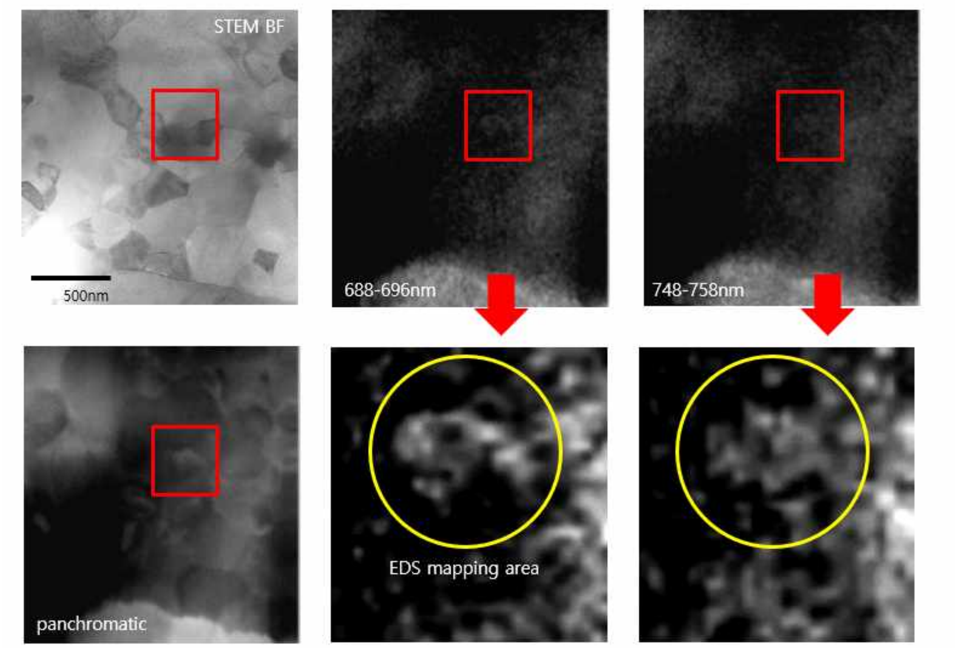

Figure 9. Monochromatic image comparison for first unknown peak and the magnified area of interests.

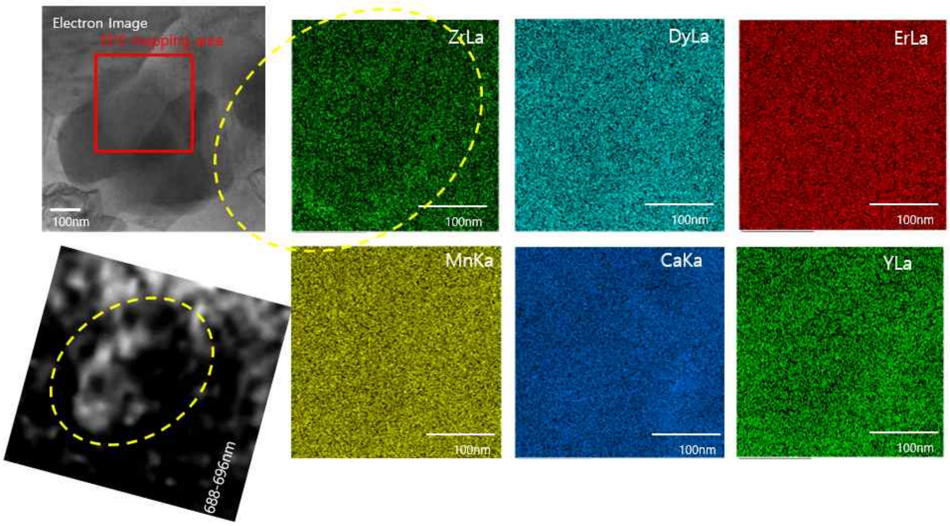

Figure 10. EDS mapping image of the interested area from fig. 9

Figure 10 shows EDS mapping image of specified region as marked. We confirmed that *Zr* elements are gathered along the grain boundary. The reason that the EDS signal is so weak is that;

First, the dopants are added less than 0.3 mol% that it is difficult to detect with EDS[5]. Second, the amount of scattering that is admitted by detector can be different with respect to different zones.

To identify the origin of second unreported peak, monochromatic image for 748-758nm was investigated. Since the magnified monochromatic image for 748-758nm, as well, shows the unique emission at the grain boundary that we conducted EDS mapping for the applicable area.

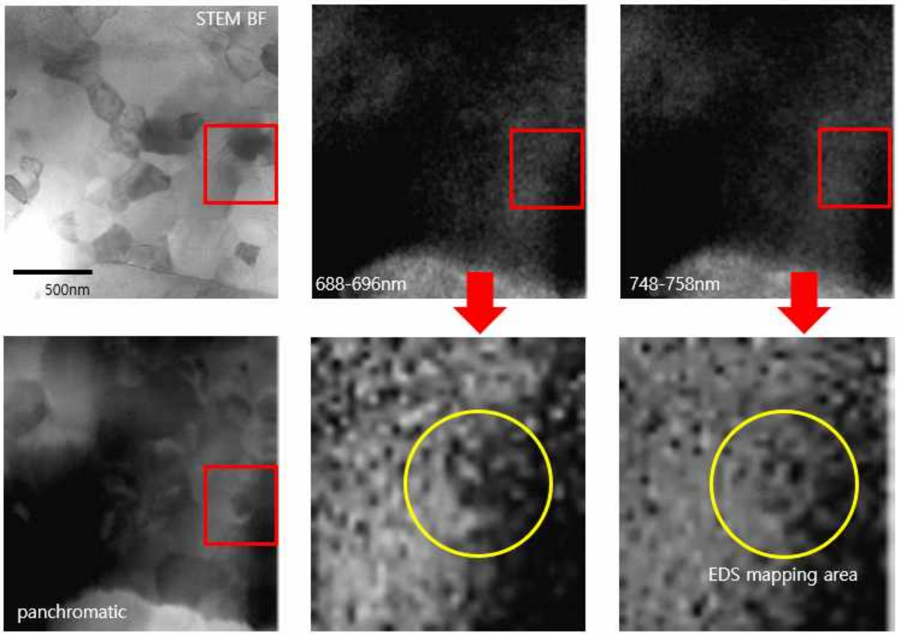

Figure 11. Monochromatic image comparison for second unknown peak and magnified area of interests.

Figure 12 shows corresponding EDS mapping images. Along the grain boundary, Yttrium ion was detected, and we could confirm that the second unknown peak was introduced by yttrium.

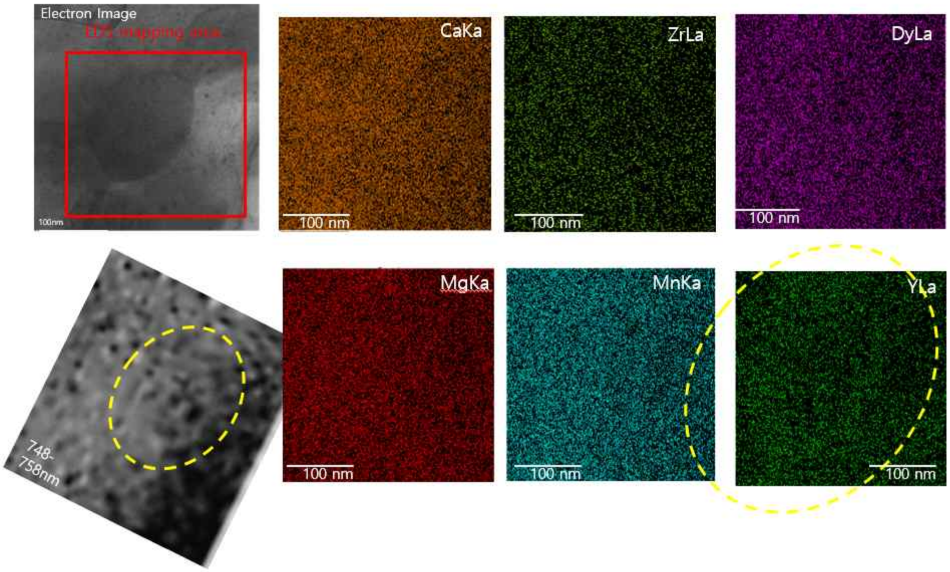

Figure 12. EDS mapping image of the interested area from fig. 10

3.3 Distribution of identified elements

Through the comparison of monochromatic images and EDS mapping data, it can be inferred that 2 newly found peaks were originated from Zr and Y respectively. As Zr has valence of 4+ as Ti^{4+}, it is suitable for isovalent substitution into Ti site. In addition, Y^{3+} ion has atomic radius of 0.9Å, which is in between Ti4+ (0.6Å) and Ba2+(1.4Å), representing that Y^{3+} ion can be substituted into both

Ba and Ti sites [5]. For these reasons, we assigned 2 newly found peaks were introduced by *Zr* and Y.

Last, we visualized not only the monochromatic images of *Zr* and Y related peaks but the defect distributions of vacancy and impurity together. This can be meaningful since it allows to visually understand where and how defects are located. Furthermore, it is possible to observe what happens after degradation of the sample; The changes in distribution, defect movement after treatments.

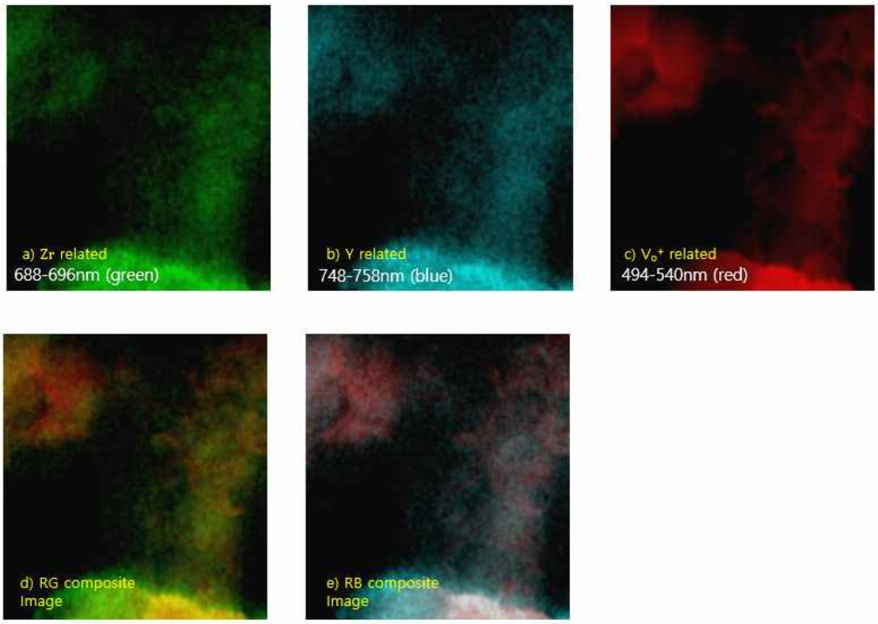

Figure 13. Monochromatic images of *Zr*, Y, V_o^+ defects and their distributions.

Chapter 4. Summary

To overcome the limitation of SEM-CL and to acquire microscopic information, defect distribution of $BaTiO_3$ dielectric for MLCC was investigated by TEM-CL. It reveals defect spectrum, types of defects, corresponding transitions and microstructural defect distributions of each types of vacancies. It is also shown that oxygen vacancy is prevailing defect in the sample. Moreover, two of unreported peaks were found, estimating the possibility of observation as resolutional advantage of TEM. Each peaks is thought to be introduced by impurities. Identification was done through the comparison of monochromatic and EDS mapping images and concluded that Zr and Y ions contributed the formation of new energy levels. Finally, we visualized defect distributions of Zr and Y ions as well.

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초록

Cathodoluminescence (CL)는 고체인 물질에서 X-ray에서부터 거의 적외선에 이르는 광자의 전자 자극에 의해 방사되는 발광이다. 따라서 이 전자기 스펙트럼에서 자외선부터 중간 적외선으로 생성 된 방사능을 CL이라고 한다. 이 CL은 많은 정보를 포함하고 있는데 대표적으로 공간적 또는 스펙트럼 적 데이터를 얻을 수 있다. 또한 CL은 $BaTiO_3$, $SrTiO_3$, $PbTiO_3$ 등의 perovskite 구조를 가진 세라믹 연구에 있어 다양한 세라믹 산화물의 효율적인 분석을 제공하므로 세라믹 연구에 일반적으로 적용되는 기술 중 하나이다[8]. 또한 CL은 light transport이나, 반도체 등을 포함하는 electronic structure of substances의 산란을 연구하는데 사용된다. 따라서 CL은 기초 연구로도 사용될 수 있을 뿐만 아니라 metrology and failure analysis와 같은 산업계와 직접적인 관련이 있는 응용 연구에 사용될 수 있는 유용한 정보를 가지고 있다.

Multilayer Ceramic Capacitor (MLCC) 시장의 성장으로 defects에 대한보다 신뢰할 수 있는 데이터가 필요시 되고, 때문에 제품의 신뢰

성은 큰 관심사가 되고 있는 상황이다. 이에 따라 MLCC에서 사용되는 $BaTiO_3$ 유전체에 관한 많은 연구가 cathodoluminescence 기법으로 수행되었지만 지금까지는 SEM (Scanning Electron Microscopy-Cathodoluminescence)-CL을 기반으로 한 연구에 치중되어 있었다.

본 실험에서는 저배율 및 저해상도에 대한 SEM-CL의 한계를 극복하여 신뢰성 있는 데이터를 얻고자 Transmission Electronic Microscopy (TEM)-CL을 사용하여 BTO 유전체를 분석하였다.

분석 결과 지금까지 보고되어 온 밴드 갭 구조와 공극의 에너지 준위를 확인하였다. 각 유형의 결함 (산소 및 바륨 공극)은 monochromatic CL 이미지를 통해 분포도 및 상대 농도를 시각화되었다. 또한 두 개의 보고되지 않은 peaks가 발견되었다. 이는 TEM의 SEM에 비해 더 좋은 해상도와 더 높은 배율을 가지고 있기 때문에 보고되지 않은 peaks가 관찰된 것으로 생각된다.

MLCC에는 많은 원소들이 신뢰성을 높이기 위하여 doping이 되기 때문에 발견 된 두 개의 보고되지 않은 peaks는 dopants로부터 생성되었다고 추정하였다. 두 개의 보고되지 않은 peaks의 원인을 확인하기 위해 monochromatic 이미지를 Energy Dispersive X-Ray Spectroscopy

(EDS) mapping image와 비교하며 조사하였다. 그 결과 peaks는 각각 Zr과 Y 원소로 인해 생성된 것으로 나타났다. 마지막으로 각각의 원소의 분포도와 상대적인 농도 또한 시각화하였다.

실험을 통해 얻은 결과는 보이지 않는 결함을 시각화하고 $BaTiO_3$ 내부의 발광 특성을 이해하는 데 도움이 될 수 있다고 생각되고 향후 절연 저하 등의 문제 시 결함의 이동 파악에 효과적일 수 있다고 생각된다.

주요어: 음극형광, 투과전자현미경, 타이타늄산바륨, 결함 분포, 적층 세라믹 콘덴서, 결정립계

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감사의 글

설렘과 꿈을 안고 학교 정문을 들어섰던 게 엊그제 같은데, 어느덧 책임감을 지고 학교 정문을 나가야 하는 시간이 왔습니다. 지난 학위기간 동안의 생활은 제가 얼마나 부족한지 많이 되돌아 볼 수 있게 해준 시간이었습니다. 부족한 제가 무사히 석사생활을 마치기까지 주변의 많은 도움이 있었기에 감사의 말을 드리고 싶은 분들이 너무 많습니다. 그 분들의 도움이 없었다면 학위를 무사히 마칠 수 없었을 것이기에 짧은 지면으로나마 감사의 마음을 전합니다.

먼저 저의 지도교수님이신 김영운 교수님께 깊은 감사의 인사를 전합니다. 연구에 대해서 아무것도 모르던 저에게 교수님께선 많은 지도를 통해 연구가 무엇인지에 대하여 많은 가르침을 주셨습니다. 또한 연구 방법 뿐만 아니라 인생에 필요한 지혜 그리고 능동적 자세 등 많은 것들을 가르쳐주셨습니다. 실수가 있어도 넓은 마음으로 받아주시고 연구자의 길을 가르쳐 주신 덕분에 늦게나마 제대로 된 연구 활동을 할 수 있었습니다. 학문에 있어 늘 열정적이셨던 모습, 마음속에 늘 새기며 조금이나마 닮아갈 수 있도록 노력하겠습니다. 앞으로도 교수님의 제자로

서 부족함이 없는 연구자가 될 수 있도록 최선을 다하겠습니다. 다시 한 번 깊은 감사드립니다.

또한 바쁘신 가운데에도 제 학위논문의 심사위원을 맡아주시고 좋은 조언을 해 주신 장호원 교수님과 홍성현 교수님께 감사드립니다. 석사 졸업 논문에 날카로운 지적과 따뜻한 조언을 해주신 덕분에 논문을 무사히 작성할 수 있어 석사 졸업을 할 수 있게 되었습니다. 이에 정말 감사드립니다.

다음으로는 우리 IEML의 실원들께 감사의 말씀 올리겠습니다. 먼저 지금은 연구실에 계시지 않지만 항상 챙겨주시고 도와주신 미향누나, 처음 연구실에 들어와 아무것도 모르는 상태의 저를 적응할 수 있게 도와준 듀오 석용이랑 위주 정말 감사합니다.

또한 IEML의 실세 용희형. 매번 밤을 샐 때마다 함께 힘들어하던 형이 있어서 이번 겨울은 덜 추웠던 것 같아요. 무엇보다 형 덕분에 평생 먹을 해산물 다 먹은 것 같아요. 항상 같이 고민해주시고 디스커션 해주시고, 힘들 때마다 옆에서 응원해 주셔서 너무나 감사해요. 영원히 IEML을 이끌어 주세요. 그리고 우리 IEML의 둘째형 원우형, IEML 베이비 이준이 육아하시느라 정신없이 바쁘실 텐데도 항상 이야기 잘 들

어주시고 연구에 대해 다방면으로 조언해주시고 도와주셔서 정말 감사합니다. 오랫동안 IEML을 지켜주세요. 3년간 내 옆자리였던 명철아. 맨날 하나하나 다 물어볼 때마다 귀찮아하지 않고 친절히 알려줘서 고마워. 남은 기간 열심히 해서 무사히 졸업하길 바래. Chen my friend. I am so glad you are graduating. Congratulations dude!! May your next adventure bring you peace. 이제 학위 시작하는 연구실 분위기 메이커 용석아. 고생해라. 못난 형들 투정 다 받아주고 궂은일 도맡아 하면서도 항상 열심히 노력하는 모습 정말 대단한 것 같아. 조금 더 고생하고 원하는 일 잘 이뤘으면 좋겠어. 올해에도 천하제일sumx대회 같이 보자. 언제한번 모두 다 같이 노랑랜드에서 회 한 접시 하면 좋겠습니다.

또한 마음잡지 못하고 방황할 때마다 힘이 되어준 명재형, 윤호형, 제윤이, 준호, 준환이, 현지, 제니, 평티모 정말 고맙습니다.

마지막으로 고생만 시킨 저를 위해 힘이 되어주시고 기도해주신 가족들께 모든 걸 바칩니다. I pray for you as you've always prayed for me. Please bless my family and protect us from harm. Give us grace to forgive, strength to overcome all the difficulties we face and keep us together when the world tries to pull us apart.

정말 감사드리고 사랑합니다.

Viva La Vida.

2019년 1월 관악에서

김 진 욱 드림