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# MICROSTRUCTURE AND DIELECTRIC STUDIES OF BISMUTH VANADATE BASED LTCC

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## ABSTRACT

**Magnesium and Copper doped bismuth vanadate with nominal composition $Bi_{1.5}Mg_{1-x}Cu_xV_{1.5}O_7$ was synthesized using classical solid state reaction method. X-ray analysis confirms the formation of monoclinic phase in the as-prepared compound. Different grain size after primary investigation reveals the influence of dopant ions on the domain structure and its microstructure. The phase composition behaviour was found to be strongly correlated with the partial substitution of heterovalent cation dopant. Energy dispersive spectra confirm the occurrence of stoichiometric elements within the synthesized compounds. $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ ceramics has low dielectric constant and higher loss values..**

**KEYWORDS :** Bismuth vanadate ceramics, Microstructure, Dielectrics, Energy dispersive spectra, LTCC

Low temperature cofired ceramics (LTCC) is a multilayer substrate manufacture technology which demands certain characteristic dielectric material to provide advantageous outcomes in the high frequency application unlike other technology. It allows miniaturization of microwave devices to reduce the size of wireless communication system (Andreas, 1977, Michael,1997). These ceramic materials have significant parameters such as enlarged usage of interconnect substrate especially in microwave applications due to their high conductivity, low dielectric loss, and cost-effective manufacturing process (Daniel I,1998). Although much work has been done in evaluating the electrical performance of various LTCC but the literature has minimal information on the bismuth substituted vanadate compounds. Bismuth based oxide materials are one of the most interesting compounds because of their unique dielectric properties and have widespread applications in sensors, transducers, memory storage devices, displays and electro-optical devices.

Bismuth oxide layered compounds with vital elements in the stack of elements gives variety of new compounds. This is one attempt to prepare Mg and Cu ions doped in the bismuth vanadate ceramics viz $Bi_{1.5}Mg_{1-x}Cu_xV_{1.5}O_7$ (x=0.2, and 0.8) using solid state reaction technique and studied their microstructure and frequency dependent dielectric behaviour.

## MATERIALS AND METHODS

$Bi_{1.5}Mg_{1-x}Cu_xV_{1.5}O_7$ (x=0.2, 0.8) were prepared using conventional solid-state reaction. $Bi_2O_3$, MgO, CuO and $V_2O_5$ (All 99.5% pure) were mixed in an agate mortar for 3 h. The mixture was calcined twice at $650^0$C for 2 h in air. The calcined powder was milled again for 4 h, sieved and compacted in circular pellets and sintered at $690^0$C for 4 hours.

The phase composition of each sample was investigated by powder x-ray diffraction (XRD, Philips, X'pert Pro PANanalytical) using *Ni*-filtered *CuK* ($\lambda$=1.5406Å) radiation between $2\theta=10^0-80^0$, with a step size of $0.02^0$ with a scan step time 0.5 having sampling rate of 5 s $step^{-1}$. The XRD pattern was analyzed by the Rietveld method using the UNITCELL program. A pseudo-Voigt function was used to generate the line-shape of the diffraction peaks. The micrograph image and energy dispersive spectra (EDS) pattern were obtained for the microstructure and elemental analysis of the polished samples using scanning electron microscope (Carl Zeiss EVO18 EDS Oxford). Dielectric measurements were performed in the frequency range of 1kHz-1MHz with an applied bias voltage of 1V using a WAYNE KERR, 43100 LCR METER controlled by a personal computer. The pellets were placed in a sample holder with a two-electrode configuration after electroding with silver. The silver paint

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was dried in an oven at $150^0$ C to remove any moisture before taking the measurements.

## RESULTS AND DISCUSSION

After calcinations at 650°C for 4 h, x-ray diffraction pattern of the $Bi_{1.5}Mg_{1-x}Cu_xV_{1.5}O_7$ revealed the single phase nature of the compound. The powder x-ray pattern was indexed based on monoclinic crystal symmetry. The lattice parameter and crystal symmetry were determined by Rietveld analysis (Shivalingayya, 2016). SEM and EDS investigation of $Bi_{1.5}Mg_{1-x}Cu_xV_{1.5}O_7$ ceramics sintered at 690°C for 4 h revealed less porous and highly dense material which are shown in Figures 1 and 2.

The variation of dielectric constant and dielectric loss of $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ ceramics with frequency at different temperatures is shown in Figures 3 and 4. $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ ceramics has low dielectric constant and higher loss values. A dielectric constant of $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ varies between 8.5-5.4 in the frequency range of 1 kHz-1 MHz at two temperatures of 50°C and 100°C. $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ ceramics has lower dielectric constant than $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ ceramics. This could be due to presence of secondary phases in $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$. Comparison of dielectric parameters of $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ are given in table 1. For both the ceramics, the dielectric constant rapidly decreases at lower frequencies and remain fairly constant at higher frequencies.

The dielectric loss for both the synthesized compounds also found to decrease with frequency and remains almost constant at higher frequency region. $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ ceramics has higher dielectric loss values than $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$. The higher value of dielectric constant at low frequency is due to the presence of all types of polarization (i.e., interface, dipolar, ionic and electronic, etc.), but at higher frequencies ($\geq 10^4$Hz), electronic polarization is the main contributor for dielectric constant.

The decrease in dielectric constant and dielectric loss with frequency is one of the features of normal dielectrics (Moulson, 2003). At high frequencies, there is no much frequency dispersion of dielectric properties for both $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ LTCC materials which is an important property for many applications (Sebastian, 2008). The dielectric loss of $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ is more than $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ and this could be due to the presence of higher concentration of CuO and secondary phase in $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$.

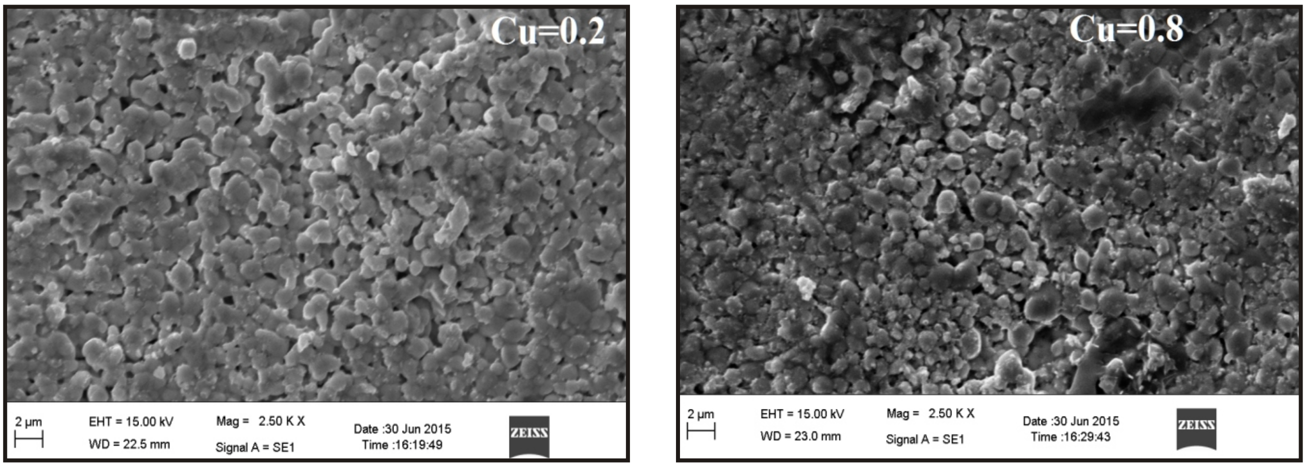


**Fig.1: Scanning electron micrographs of ($Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}$) $O_7$ and ($Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}$) $O_7$ LTCC**

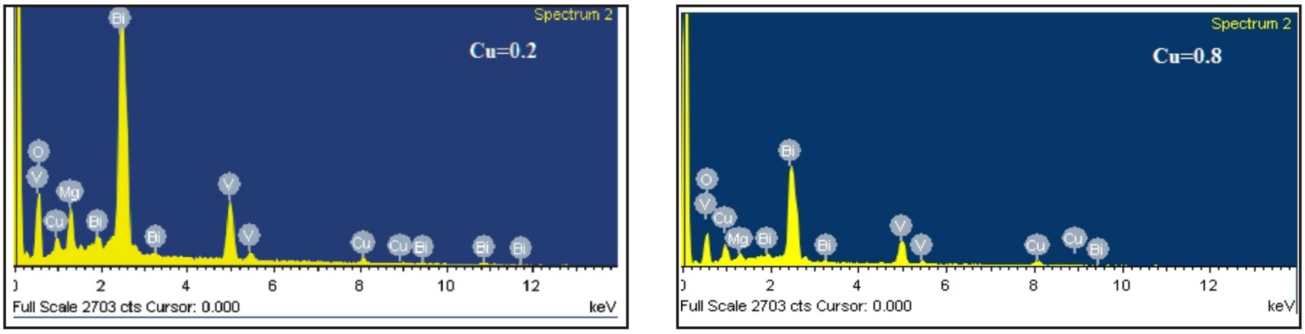


**Fig. 2: Energy dispersive spectra of $(Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5})O_7$ and $(Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5})O_7$ LTCC**

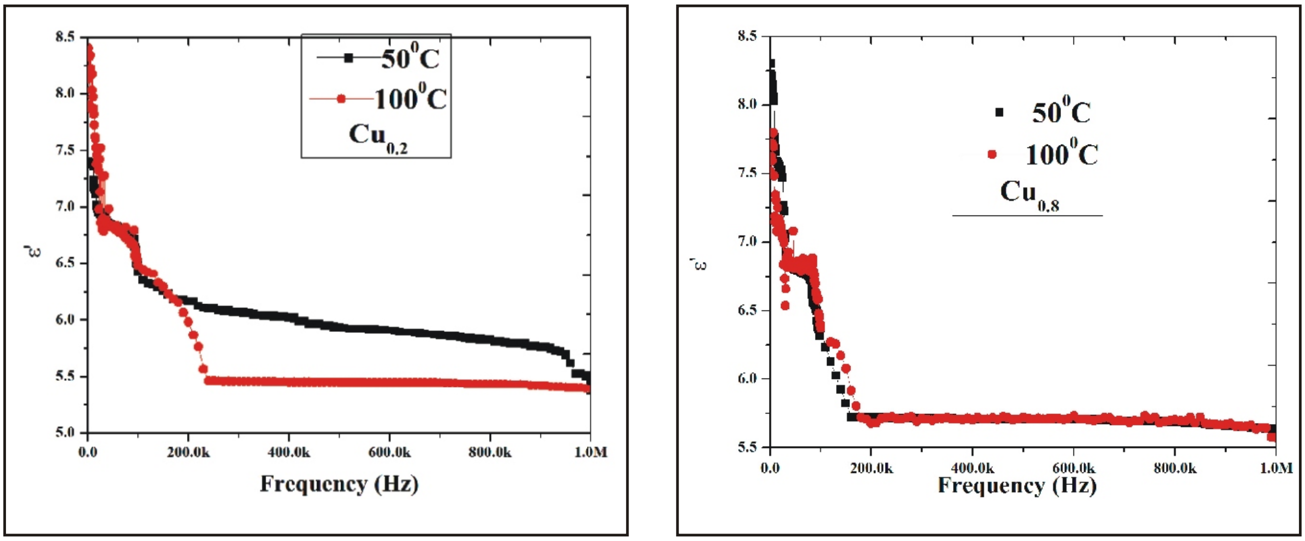


**Fig. 3: Frequency dependent dielectric constant behavior at of (a) $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and (b) $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ LTCC at different temperatures**

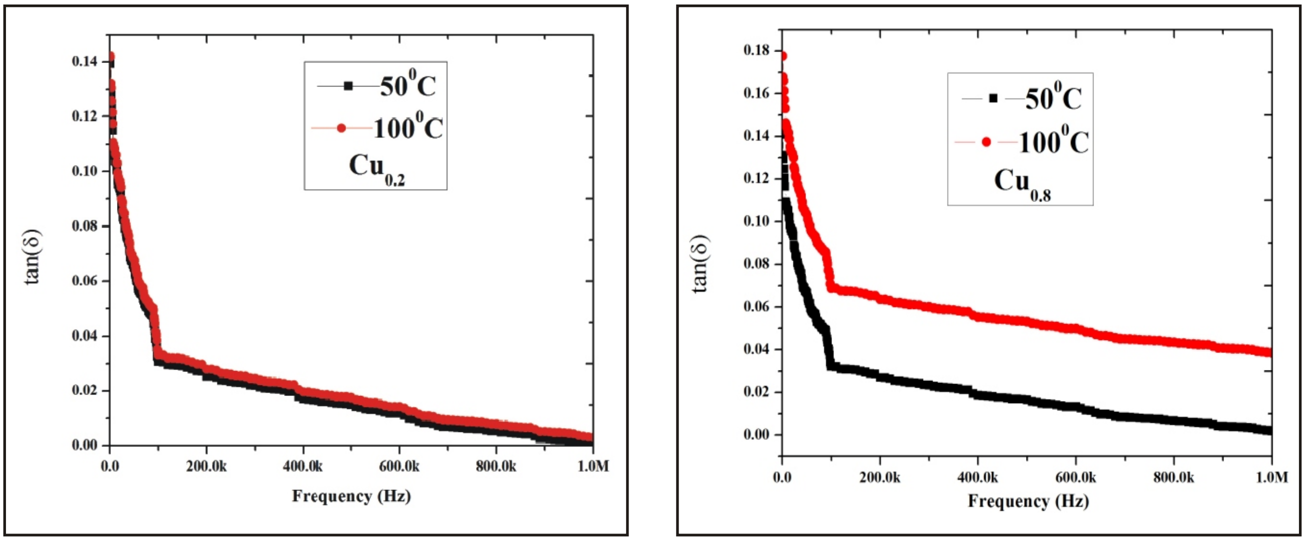


**Fig. 4: Frequency dependent dielectric constant behavior at of (a) $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and (b) $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ LTCC at different temperatures**

**Table1: Comparison of dielectric properties of $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$**

| Ceramic compound | Test Frequency | Dielectric constant (ε') | | Dielectric loss (tanδ) | | Sintering temperature and |
|---|---|---|---|---|---|---|
| | | $50^0C$ | $100^0C$ | $50^0C$ | $100^0C$ | time |
| $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ | 1MHz | 5.44 | 5.39 | 0.016 | 0.030 | $690^0C$; 4hrs |
| $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ | 1MHz | 5.60 | 5.57 | 0.020 | 0.039 | $690^0C$; 4hrs |

## CONCLUSIONS

$Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ and $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ compounds were produced from mixed oxide technique and their dielectric properties were investigated as a function of frequency at different temperature. XRD patterns revealed pyrochlore phase in $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ whereas SEM results displayed CuO-rich phase in addition to pyrochlore phase in $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$. $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ pyrochlore has higher dielectric constant and higher dielectric loss values than $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ at temperature 50°C and 100°C. $Bi_{1.5}Mg_{0.2}Cu_{0.8}V_{1.5}O_7$ pyrochlore LTCC exhibits less dielectric constant and lower dielectric loss than $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$ due to the existence of secondary phases in $Bi_{1.5}Mg_{0.8}Cu_{0.2}V_{1.5}O_7$.

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