NiCoZn Ferrite Sintered With BBSZ Glass For LTCC Based Inductive Devices

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Abstract

Spinel ferrite [$Ni_{0.2}Co_{0.3}Zn_{0.5}Fe_2O_4$] material has been synthesised for the use with Low Temperature Cofired Ceramic (LTCC) substrate material for embedded magnetic devices. Integration of such magnetic devices however, requires matching sintering temperatures of both materials. Considering the high sintering temperatures of ferrite materials compared to that of LTCC (≤900°C), this imposes the development glass-ferrite composites having requisite magnetic properties such as coercivity, saturation magnetization and remanent magnetization. The NiCoZn ferrite is synthesized by combustion synthesis method with neutral precursor solution. Structural and magnetic properties of the calcined ferrites were studied using vibrating sample magnetometer (VSM), respectively. The VSM result shows coercivity, saturation magnetization and remanent magnetization close to ~80 Oe, ~72 emu/g and ~5 emu/g respectively. Both properties promise as suitable for magnetic device applications. Experiments are carried out to optimize glass percentage and sintering temperature so as to achieve high densification of NiCoZn ferrite suitable for use in LTCC process. The ferrite material sintered with BBSZ glass shows relatively good sintered density (~85-90%) and electrical properties, permeability constant ~10 thus emphasizes use in LTCC based magnetic devices for high performance applications viz. fabrication of embedded inductors operating at RF frequencies

Keywords: *NiCoZn ferrite, combustion synthesis, embedded magnetic devices, Sintering Composites*

1. Introduction

The technologies that could be explored are thin-film, polymer and ceramic type electronic package preparation techniques. Among these, the technology with far greater possibility of integration and ease, is the low temperature co-fired ceramic (LTCC) technology [1]. LTCC is a glass-ceramic, multilayer circuit, and electronic package preparation process that has great advantage over the other, more conventional electronic packaging technologies. These circuits already offer resistive and capacitive materials that could be integrated with the host materials of low-loss dielectrics and can be interconnected using via and three dimensional conductor networks. The LTCC materials, therefore, are quite suitable for microwave circuit fabrication. Clearly, LTCC shows promise for true integration of microelectronic circuits along with passive components and mostly in HF and microwave circuits and modules from 1 GHz to 40 GHz, and some even up to 100 GHz frequency range [2]. Selecting Materials for magnetic components, such as, inductors and circulators are challenges for integrated inductors in LTCC dielectrics are low range of inductance. Higher inductance is possible with materials having permeability larger than one. Ferromagnetic metals and ferrites are seen as an option. It is noted that, ferrites are preferred for both applications in RF/microwave frequencies. The properties which have made ferrites so useful at these frequencies are: permeability and resistivity. Ferrites are popular as RF multilayer chip inductors (MLCI). High performance MLCIs are available commercially and are widely used in substrate boards such as LTCC. Due to advancement in low temperature synthesis of ferrites, the focus has now turned to integrate these materials with LTCC.

A very few reports are available on the integration of ferrite materials with LTCC system. It is clear that NiCuZn and NiCoZn ferrites[3,4] are the most explored material system for the integration with LTCC, mainly due to its low temperature densification (~900 ^{o}C) capability. The densification at temperature around 900^{o}C is enhanced by the use of well-known low temperature sintering aids such as Bi_2O_3 or low melting reactive BBSZ glass (Bi_2O_3-B_2O_3-SiO_2-ZnO glass) which is widely used in LTCC [5,6].

NiCoZn ferrite [$Ni_{0.2}Co_{0.3}Zn_{0.5}Fe_2O_4$] material has been prepared with an aim of using with Low Temperature Cofired Ceramic (LTCC) substrate material for embedded magnetic devices such as inductors, circulators, isolators, etc. Such integration, however, requires matching sintering temperatures of both materials. The NiCoZn ferrite was synthesized by combustion synthesis with neutral precursor solution [7-9]. Structural and magnetic properties of the calcined ferrite were studied using x-ray diffraction (XRD) and vibrating sample magnetometer (VSM), respectively.

2. Experimental work

2.1 Synthesis of (NiCoZn) Fe_2O_4 Spinel Ferrite:

NiCoZn ferrite powders were obtained by combustion synthesis method with neutral precursor solution with aim of using it with LTCC technology. The synthesized powders were characterized using x-ray diffraction (XRD) and vibrating sample magnetometer (VSM) in order to study their structural and magnetic properties. Preparation of ferrite pellets sintered at LTCC temperatures, viz. 900 ^{o}C and characterization for performance as magnetic material, which includes measurement of resistivity, density and HF permeability. Fig. 1 shows the experimental work of synthesis.

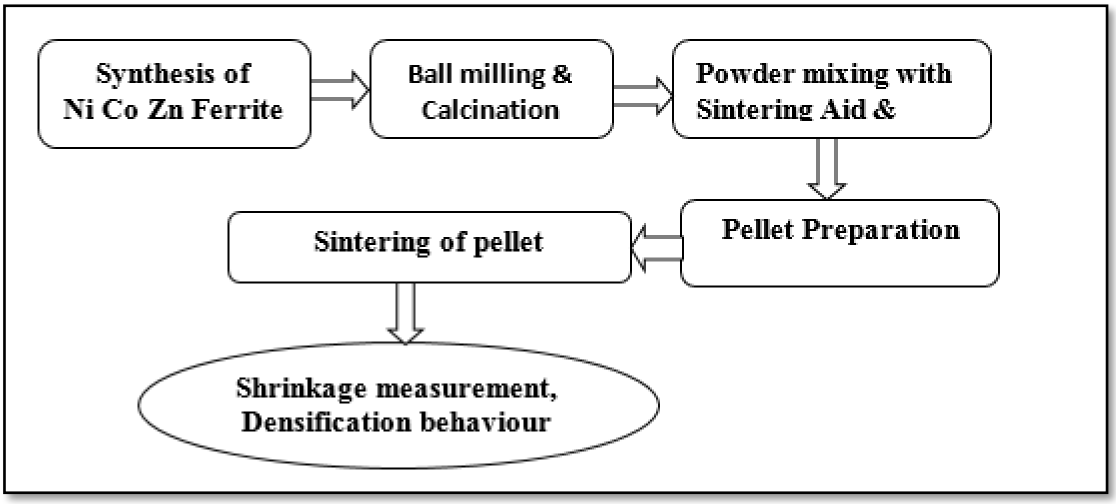

Fig. 1: Synthesis of NiCoZn ferrite powders

2.2 Pellet Preparation:

The powder as-obtained from the above heat-treated conditions (calcination) is mixed with δ-amount of BBSZ glass (where, δ = 2, 5 and 10wt %) & 7% polyvinyl alcohol (PVA) solution as a binder. The powder is mixed in an agate mortar & pestle for 1 h. The resultant powder was then uniaxially pressed at a pressure below 100 MPa to fabricate green circular discs (diameter 10 mm). The pellets were sintered in an air atmosphere at temperatures 850, 875 & 900 ^{o}C for 2 hrs and cooled in the furnace. The pellets prepared by above methods are subject to various physical, structural and magnetic (property) investigations. Following measurements are noted during the optimization of shrinkage & density of NiCoZn ferrite.

3. Result and discussion

3.1 Vibrating Sample Microscopy (VSM):

The magnetic properties, such as, saturation magnetization (σs), coercivity (Hc) and permanent magnetization (σr) are important in determining the efficiency of magnetic material. These magnetic properties of synthesized NiCoZn ferrite are obtained from MH curve measurements carried out on vibrating sample magnetometer (VSM) at room temperature with a maximum applied field of 10 kOe. Figure 2 presents the M-H curves of NiCoZn ferrite prepared using ceramic route and calcined for different temperature and time cycles.

The saturation magnetization and coercivity of the powder sample were 70 emu/g to 75 emu/g and 1350 Oe to 3500 Oe, respectively, varying with the starting pH conditions and calcination temperature. These properties depend largely on the preparation conditions, resulting in variation in particle sizes and consequently the magnetic properties. Fig. 2 (a) and (b) shows the Magnetization of $NiCoZnFe_2O_3$ plot of $NiCoZnFe_2O_3$ material.

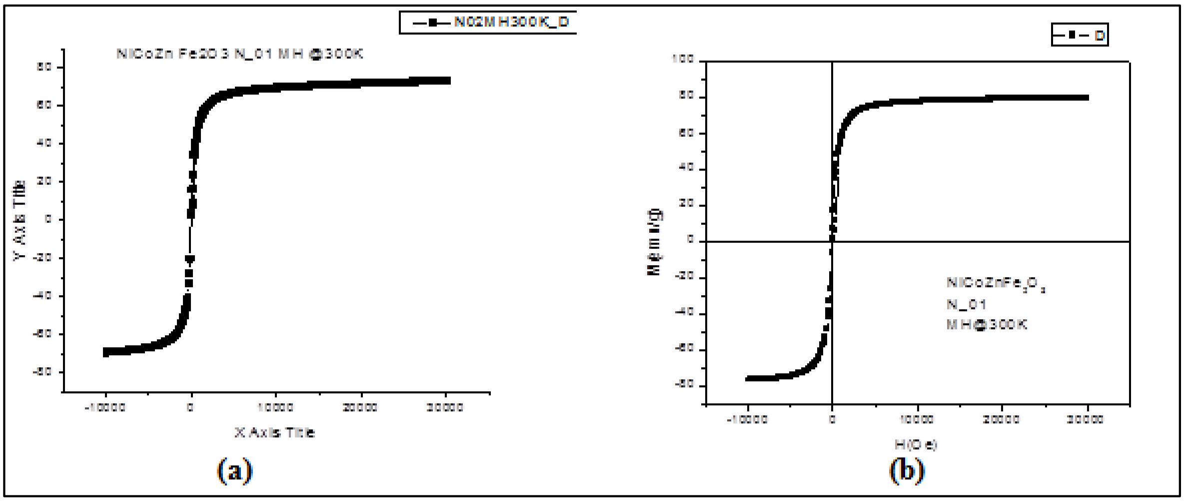

Fig. 2: (a) & (b) Magnetization of NiCoZnFe_2O_3 material Calcined at 900°C

3.2 Dilatometer for Sintering Temperature:

A pellets is prepared with 6 mm diameter & 5 mm thickness with the said composition and applied for dilatometer testing for sintering temperature, it is observed that, the pellet with higher percentage of BBSZ sinters at 800 temperature. Fig. 3 reveals the dilatometer Curve for NiCoZn with BBSZ.

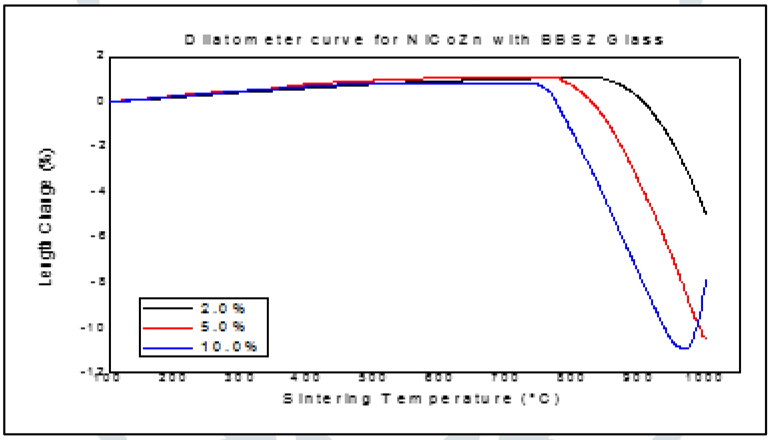

Fig. 3- Dilatometer Curve for NiCoZn with BBSZ

3.3 X-ray Powder Diffraction:

The synthesized NiCoZn ferrite materials annealed at elevated temperature with a live time results in polycrystalline bulk material. The identification of the crystalline phases in spinel ferrite materials is routinely possible with X-ray powder diffraction (XRD) technique. The XRD patterns of NiCoZn [$Ni_{0.2}Co_{0.3}Zn_{0.5}Fe_2O_4$] ferrite material are shown in Fig. 4, for the powder samples prepared using solution combustion method. The diffraction peaks establish beyond doubt the single-phase cubic spinel structure and do not show any additional diffraction peaks, including those of residual reactants or raw material. All peaks can easily be indexed to (230), (311), (222), (400), (422), (333), (440) with highest intensity peak observed at (311) planes of the polycrystalline spinel phase. The average crystallite size for the sample calcined at 900 ºC for 2 h.

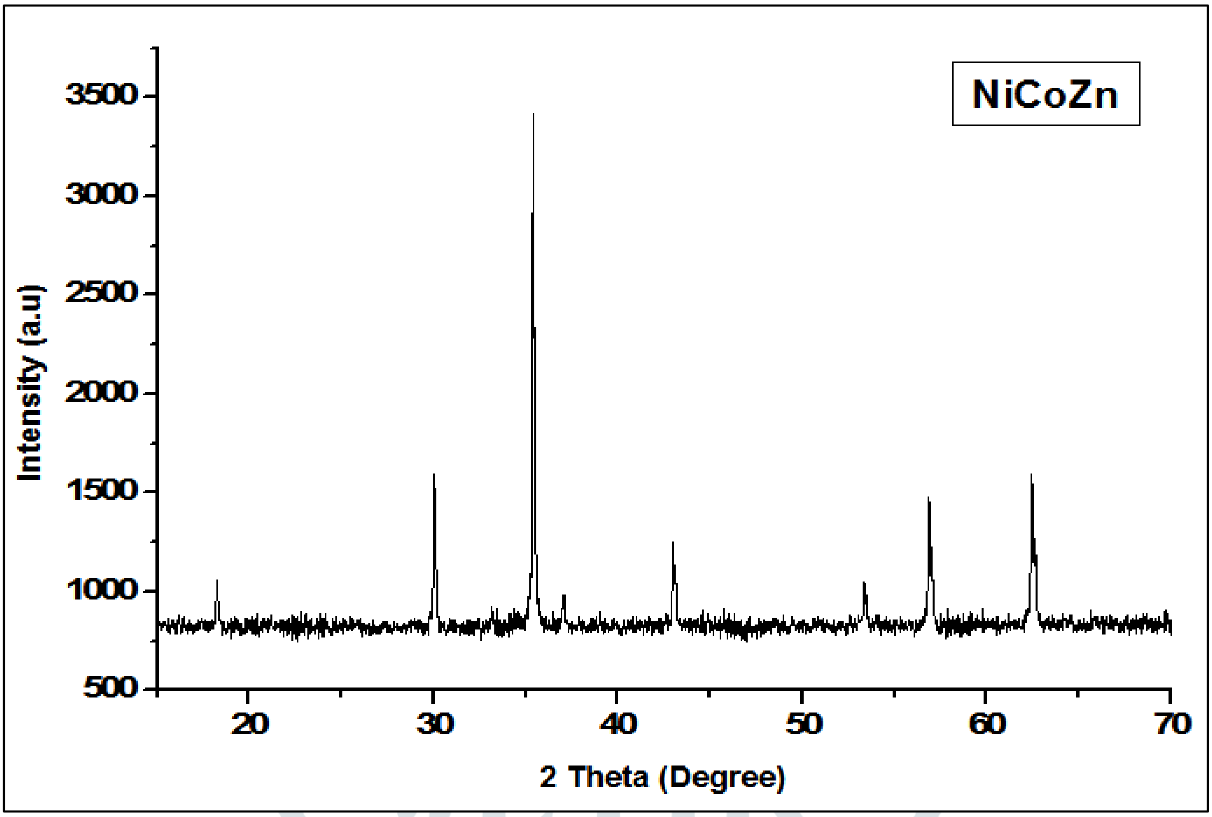

Fig. 4: XRD plot of NiCoZn

From XRD it is observed that, the prominent peak is present at 35.41° and indicating (222) plane. Using Debye sheerer formula crystallite size was estimated and it was found to be 65.28 nm. From XRD analysis it is confirmed that, using solution combustion method NiCoZn nanoparticles could be synthesis.

Table 1: Pellet measurement of NiCoZn

BBSZ (%)	**Sintering Time (Hrs)**	**Temp**	**After sintering**		**Before**		**% shrinkage**	
			OD	**ID**	**ID 3.5**	**ID 3.6**		
2	2	925	7.61	3.26	3.5	3.6	6.857143	9.444444
4	2	925	7.58	3.18	3.5	3.6	9.142857	11.66667
6	2	925	6.79	2.9	3.5	3.6	17.14286	19.44444
8	2	925	7.48	3.16	3.5	3.6	9.714286	12.22222
10	2	925	7.45	3.19	3.5	3.6	8.857143	11.38889

Conclusion:

The formation of single phase spinel structure was confirmed by XRD. The VSM result shows coercivity, saturation magnetization and remanent magnetization close to ~80 Oe, ~72 emu/g and ~5 emu/g respectively. The ferrite material sintered with BBSZ glass shows relatively good sintered density and electrical properties, thus emphasizes use in LTCC based magnetic devices for high performance applications.

- **Shrinkage:** shrinkage matching with LTCC at 5% composition of BBSZ Glass and 900°C sintering temperature
- **Densification:** 80 to 82 % density obtained at the 4-5% composition of BBSZ Glass and 900°C sintering temperature

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