EFFECT OF CU ON THERMOELECTRIC PROPERTIES AND ELECTRONIC BAND STRUCTURE OF INKJET PRINTED ZN$_x$Fe$_2$O$_4$ THIN FILMS

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Abstract

In this paper, Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films were deposited by using inkjet printing and Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ bulk pellets were synthesized through solid state method. Multiple print cycles were required to deposit homogeneous Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films. The obtained samples were characterized by X-ray diffraction (XRD), electrical conductivity, Seebeck coefficient and thermal conductivity. The XRD results confirmed the formation of cubic spinel structure of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films and pellets. The electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ (x=0.0) thin films sintered at 400 °C (1.185x10$^{-3}$ S/cm) had the higher values. The electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films was about 11% higher compared to Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets. The electronic band structure shows Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ is an indirect band gap material. The Fermi level of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ was shift downward to the valence conduction band. It indicated Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ is a p-type semiconductor. Seebeck coefficient of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films and pellets remained positive, confirming charge transport by hole carries. The presence of Zn served to decrease thermal conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ by 8 W/mK as Zn content increased from 0 to 1. The similarity observed in the change of properties might indicate that similar mechanisms are dominant in both the Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ bulk pellets and the thin films.

Keywords: Inkjet printing, electronic band structure, thin films, thermoelectric, p-type semiconductor

I. Introduction

Materials formed with a thickness that in the range of micrometers or less are called thin films [VI]. The thickness of thin film is typically less than 50 µm thick. The defined thickness is highly dependent on the application. These films are different from the bulk materials in terms of properties and vary according to the
condition of deposition and the thickness of the films. The general properties of materials are determined by the atomic compositions [XII]. The unique properties of thin films have made possible various applications such as hard coatings, wear resistant films and optical devices [XI]. Since then, thin films have become a major influence in the electronic arena. The primary advantages and influence of thin films in the electronics field include a faster response time, lower power requirements and smaller dimensions [IX]. Thin films have been fabricated by different deposition methods such as thermal evaporation, pulsed laser deposition, spin spray plating and electron beam evaporation [VI-XI]. There is an interest in the development of thin films due to the downsizing of the electronic components. However, one of the main drawbacks of research into thin films is the high cost of fabrication [V]. The inkjet printing process is a low cost alternative method to fabricate thin films. The inkjet printing method has the advantages of easy operation, simple equipment and the ability to pattern directly [XV]. This paper attempts to solve the issue by using commonly available inkjet printers to fabricate thin film of zinc substituted copper ferrites ($\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$).

Zinc substituted copper ferrite widely used for various applications such as radio frequency coils, transformers cores, rod antennas and magnetic cores of read-write heads [VIII, III]. The properties of $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ depending on the concentration of Cu and Zn in the ferrites [IV, II]. The possibility of mixing metals in ferrite with different composition becomes attractive. The synthesis of $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ have been reported by various researcher. Samadashvili et al. [III] prepared $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ ($x = 0.2, 0.4, 0.6,$ and $0.8$) solid solutions from appropriate mixtures of $\text{Fe}_2\text{O}_3$, $\text{ZnO}$, and $\text{CuO}$ by a conventional ceramic route and performed their calorimetric studies. Yue et al. [XVI] used the sol–gel auto-combustion technique to synthesis nano-sized Mg–Zn–Cu ferrite owing to the fact that ferrite nanoparticles can be formed directly from auto combustion of dried gel in air. In this paper, inkjet printing was used to deposit $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ thin films. The magnetic, dielectric and elastic properties of mixed Cu–Zn ferrites have been studied by various researchers. Manikandan et al. synthesized Cu–Zn ferrites ($\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ ($x = 0.0, 0.1, 0.2, 0.3, 0.4$ and $0.5$)) by microwave combustion method and studied on their optical and magnetic characteristics [I]. The structural, magnetic and optical properties of Zn-Cu ferrites have been widely reported [III] but the information regarding the thermoelectric properties of Zn-Cu ferrite is scarce.

Thermoelectric materials are capable of converting a heat into electrical energy, making them interesting for applications within power generation. One problem that limits the areas of application for thermoelectric devices is the efficiency, which is typically below 10 % for the best thermoelectric materials [II]. Due to the low efficiencies, large scale usage has sometimes been believed to be limited to vehicle exhaust heat recovery. Another problem is that many of the best thermoelectric devices are dependent on toxic and expensive materials that are limited in supply. Devices made from this scarce material would have limited potential for widespread used [IV]. Therefore, commonly used materials would be the focus for this study. There are very few reports on the thermoelectric properties of $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ in the

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literature. The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) thin films prepared through inkjet printing and the thermoelectric properties were studied in this work.

II. Materials and Methods

The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) \( (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) \) bulk pellets were prepared through solid state method. Analytical grade reagents were used. Appropriate amounts of ZnO, CuO and Fe\(_2\)O\(_3\) were prepared and shaped into bulk pellets with 3 mm thickness and 15 mm in diameter. The bulk pellets were sintered at 3 different temperatures \( (800 \, ^\circ\text{C}, 900 \, ^\circ\text{C}, 1000 \, ^\circ\text{C}) \) with oxygen for 4 hours. The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) bulk pellets were characterized for density, phase analysis, electrical conductivity, Seebeck coefficient, thermal conductivity. The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) \( (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) \) thin films were prepared by ink-jet printing. Analytical grade reagents were used. Appropriate amounts of \( \text{Cu(NO}_3\text{)}_2 \) and \( \text{Zn(NO}_3\text{)}_2, \text{Fe(NO}_3\text{)}_3 \) were used to prepare solutions based on the molar ratios of \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) \( (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) \). The solution was injected into ink cartridge of the printer (Canon pixma iP4810, Japan) for printing. The solution usage was determined by measuring the cartridge weight every 5 print cycles and varying the printed colours. After printing, the samples were sintered at 200 °C, 300 °C and 400 °C in oxygen for 4 hours with a heating rate of 10 °C/min. The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) thin films were characterized for thickness, phase analysis, electrical conductivity, Seebeck coefficient, thermal conductivity.

The surface morphology of the printed films was characterized through optical microscope (Nikon eclipse optical microscope ME600). The phase analysis was carried out through X-ray diffraction (XRD Shimadzu 2000) with Cu-K\( \alpha \) radiation source \( (\lambda = 1.5418\text{Å}) \) from 20° to 100° with scan rate 2 °C/min. The electrical conductivity measurement was carried out using a Keithley’s source measure unit (Model SMU 236) based on ASTM F42-02. The thermal conductivity was measured at room temperature according to ASTM C177-13. The Seebeck coefficient was carried out through differential method at room temperature [X]. The band structure of \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) were calculated based on the density functional theory (DFT) Kohn-Sham equations through ABINIT program [XIV]. ABINIT program is open source software used to compute band structure and ground state properties.

III. Results and Discussion

The \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) thin film was successfully deposited onto glass substrate using inkjet printing method with several repeated printing. The surface profile of the \( \text{Zn}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4 \) \( (x = 0.6) \) thin films after several print cycles was shown in Fig 1. The uniformity of print film was increased with an increasing print cycles. It can be observed that there was discontinued printed film formed after 10 times print cycles as shown in Fig 1(a). The gap between the discontinued films was filled with print cycles increased to 30 times. Homogenous film was obtained with 50 print cycles. The amount of dispensed solution was approximately 0.2 g by weighing the cartridge weight loss. Similar trend was observed for all the inkjet printing thin films. No visible change was observed if increased beyond 50 print cycles. Segregation of the compounds was not observed probably due to the small amounts of solution being...
deposited at each printing pass. Fig 2 show the cross sectional image of inkjet printing Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ (x = 0.6) thin films.

Fig. 1: Surface profile of the Zn$_{0.6}$Cu$_{0.4}$Fe$_2$O$_4$ thin films

Fig. 2: Inkjet printing Zn$_{0.6}$Cu$_{0.4}$Fe$_2$O$_4$ thin films

Fig 3(a) show the XRD pattern of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) pellets sintered at 800 °C. The sample peaks were identified as the plane reflection of cubic spinel phase based on JCPDS data file ZnFe$_2$O$_4$ (No. 22-1012) and CuFe$_2$O$_4$ (No.25-0283). The peaks were slightly shifted to the left ($\theta$) when increasing Zn content. There was no change in the peak intensity and position with increasing sintering temperatures. Fig 3(b) show XRD pattern of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) thin films sintered at 200 °C. The peaks detected (220) and (311) were identified as plane reflection of zinc copper ferrite according to the JCPDS data file of ZnFe$_2$O$_4$ (No. 22-1012) and CuFe$_2$O$_4$ (No.25-0283). Fewer XRD peaks were detected for thin film samples compared to bulk pellet samples probably due to the low dimensions and crystallinity of thin films [XIII, VII].

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The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellets and thin films decreased with increasing Zn content as shown in Fig 4. Fig 4(a) shows the electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellets (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) sintered at different temperature (800 °C, 900 °C, 1000 °C). The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (x = 0.0) pellet reached the highest values (1.031x10^{-3} S/cm). The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ decreased might be due to addition of Zn attributed to an decrease in Cu [XVII]. The conduction mechanism in Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ was due to the interaction between Cu-Cu and Fe-Fe [XVII]. The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (x=1.0) was the lowest (0.444x10^{-3} S/cm). The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ slightly increased (0.02x10^{-3} S/cm) when sintering temperature increased from 800 °C to 1000 °C. This might be due to higher density of the samples.

Fig. 3: XRD pattern of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (a) pellets (b) thin films.

Fig. 4: Electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (a) pellets (b) thin films.
Fig 4(b) shows the electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films with different amounts of Zn (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0). The electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ (x=0.0) thin films sintered at 400 ºC (1.185x10$^{-3}$ S/cm) had the higher values. The electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films was about 11% higher compared to Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets. This might due thin films had the advantages of faster response time compared to the bulk materials [II]. The electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films increased with increasing sintering temperature due the particle coalescence. The similarity in test results between the bulk pellets and the thin films might indicate that addition of Zn attribute to decrease in electrical conductivity.

![Image](image.jpg)

**Fig. 5:** Electronic band structure of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$

Fig 5 shows the electronic band structure of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$. The band structure shows that Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ is indirect band gap material with the valence band maximum (VBM) at M and conduction band minimum (CBM) at A. The Fermi level of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ was shift downward to the valence conduction band. It indicated Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ is a p-type semiconductor. The calculated band gap at point was 2.205 eV for CuFe$_2$O$_4$. Fig 5(b)-(f) shows the calculated band structure of Zn$_{0.2}$Cu$_{0.8}$Fe$_2$O$_4$ (2.213 eV), Zn$_{0.4}$Cu$_{0.6}$Fe$_2$O$_4$ (2.224 eV), Zn$_{0.6}$Cu$_{0.4}$Fe$_2$O$_4$ (2.237 eV),Zn$_{0.8}$Cu$_{0.2}$Fe$_2$O$_4$ (2.242 eV) andZnFe$_2$O$_4$ (2.249 eV). The band gap of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ slightly increased with increasing Zn content. The electrical conductivity results as shown in Fig 4 were similar to the calculated band gap where electrical conductivity of Zn$_x$Cu$_{(1-x)}$Fe$_2$O$_4$ decreased with increasing Zn content.
Fig 6: Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ (a) pellets (b) thin films.

Fig 6(a) shows the Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets sintered in oxygen. The Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets increased with increasing Zn content. The Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets sintered at 1000 $^\circ$C in oxygen was 21.191 µV/K ($x = 0.0$) increased to 25.451 µV/K, 26.891 µV/K, 28.774 µV/K, 29.766 µV/K and 31.666 µV/K when Zn content increased to 0.2, 0.4, 0.6, 0.8 and 1.0 respectively. The observed increase in the Seebeck coefficient might be due to decrease in electrical conductivity carrier concentration [XVII]. The Seebeck coefficient of all Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets remained positive, confirming charge transport by hole carries. The increase of sintering temperature (800 $^\circ$C to 1000 $^\circ$C) served to increase Seebeck coefficient by 1 µV/K. Fig 6(b) shows the Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films sintered in oxygen. The presence of Zn content served to increase Seebeck coefficient by 10 µV/K with increasing Zn content from 0 to 1. Similar result was observed with the Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellets. The Seebeck coefficient of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ thin films sintered at 200 $^\circ$C in oxygen was 6.361 µV/K ($x = 0.0$) increased to 16.724 µV/K ($x = 1.0$). This was similar to the test results of Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ pellet. The similarity observed in the change of properties might indicate that similar mechanisms are dominant in both the Zn$_{x}$Cu$_{(1-x)}$Fe$_2$O$_4$ bulk pellets and the thin films.
Fig. 7: Thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (a) pellets (b) thin films.

Fig 7(a) shows the thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellets decreased with increasing Zn content. The thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellet sintered at 1000 °C was 19.163 W/mK (x = 0.0) decreased to 16.902 W/mK, 14.578 W/mK, 12.576 W/mK, 11.483 W/mK and 11.331 W/mK when Zn content increased to 0.2, 0.4, 0.6, 0.8 and 1.0 respectively. The thermal conductivity slightly increased when increasing sintering temperature. The Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellets show higher thermal conductivity when sintered at 1000 °C. This might be due to the improvement of density as the sintering temperature increased as shown in density results [XIII]. Fig 7(b) shows the thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ thin films. The presence of Zn served to decrease thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ thin films by 8 W/mK as Zn content increased from 0 to 1. The thermal conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ thin film sintered at 400 °C shows higher value of 17.423 W/mK (x =0.0) decreased to 15.162 W/mK, 12.838 W/mK, 10.836 W/mK, 9.743 W/mK and 9.443 W/mK when Zn content increased to 0.2, 0.4, 0.6, 0.8 and 1.0 respectively. Similar results was observed for Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellet and thin films.

IV. Conclusion

The Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) thin films have been successfully deposited using inkjet printing and Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) pellets prepared through solid state method. A minimum 50 print cycles were required to deposit homogeneous films of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$. The XRD results show single cubic spinel structure of Zn-Cu ferrite was obtained for thin films and pellets. The electrical conductivity of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ pellets and thin films decreased with increasing Zn content. The conduction mechanism in these materials is mainly due to the interaction between Cu–Cu and Fe–Fe. The band gap of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ slightly increased with increasing Zn content. The presence of Zn content served to increase the band gap of Zn$_x$Cu$_{1-x}$Fe$_2$O$_4$ by 0.044 eV as Zn content increased from 0.

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to 1. The Seebeck coefficient of $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ thin films and pellets increased with increasing Zn content. The thermal conductivity of $\text{Zn}_x\text{Cu}_{(1-x)}\text{Fe}_2\text{O}_4$ thin films and pellets decreased with increasing Zn content. The similarity in thermoelectric behavior indicates that ink-jet printing was able to replicate the properties of the bulk materials in the thin films.

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