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Comparative Study of Seebeck Constant of Cuznferrite Product between Analysis of flow injection Synthesis and data Xrd-Rieltvelt Rifenement

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***Abstract**: The degree inversion ferrite of $Cu_{(1-x)}Zn_xFe_2O_4$ has been studied by using of the Rieltvelt refinement of XRD data method. Generally give a good result, then it may be used as a reference of the comparative study. The Flow Injection Synthesis-FIS is one of method forming of iron oxide such as ferrite material use principle thermodynamic then either the activation energy, entropy or Seebeck constant of material may be obtained well. The $Cu_{(1-x)}Zn_xFe_2O_4$ is one of may obtain entropy cause un-stable substance was marked by trend exchange of entropy. The entropy exchange may give free charge in the substance and give Seebeck of material exchange. This study found that there is a difference in entropy between the FIS method Rielvelt refinement XRD method. The entropy as result FIS calculation less then XRD Rieltvelt refinement method reveal the chemical reaction at FIS not yet finish and still continuing until powder performed.*

***Keywords:** co-precipitation, ferrite, non-isothermal process, crystallization, , data logger acquisition system, flow injection synthesis-FIS, inversion degree .*

I. Introduction

Ferrites have been studied since 1936. They have an enormous impact over the applications of nano magnetic particles-NMP has opened the door for completely new technologies [1]. The ferrites NPM behavior have been revealed since Robert Kaiser at Avco-Nasa Corporate 1961 explore of ferrofluid successfully [2] at room temperature, can widely use depend on their chemical compose and the range of particle size. The Iron oxides in nano-scale have exhibited great potential for their applications as catalytic material [3,4] was recognized as arrange more NPM.

A number of scientists have shown that a certain amount of iron oxides include ferrite material have properties as a semiconductor and thermoelectric due to instability of the ferrite material by energy infiltration surround. [5,6].

Based on the principle of formation reaction with Ferrite the deposition method (co -precipitation) which requires the presence of Fe 2 + ion pair and Fe 3 + ions , while the Fe $^{2+}$ ions should be on the site of octahedral , then the distribution of Ferrite cations .

Cu-Zn Ferrite equation can be expressed. .

Unstable of the certain ferrite can be increased by surround energy such as; sun energy, surround temperature, pressure and etch.

The possibility of preparing ferrite in the form unstable material can be done by direction of material fraction especially irons have tree and divalent.

One of important ferrite is magnetite-$Fe^{2+}Fe_2{}^{3+}O_4$ or Fe_3O_4 currently as a large field of research due to the fascinating properties associated with the coexistence of ferroes ferric cations. The cation of iron divalent – Fe^+ may compose by other cation metal divalent such as Cu^{2+}, Zn^{2+},Ni^{2+} and etc. Every variant as product of combine with divalent metal may give a new ferrite material that have new properties or new behavior then each composition either kind of divalent metal or mass of metal will give different product.

Composition between Cu^+ and Zn^{2+} to Fe_2O_4 where radius of Zn^{2+} larger than Cu^{2+} will give to stability behavior exchange where stability of the substance will change to be lower due to micro structure distortion of the substance.

An interesting of the unstable of ferrite is the dependency ferrite degree inversion, where the range value of inversion degree between 0 and 0.5.

The preparation of the especially ferrite may be arranged using of the optimum composition of tree valent of divalent iron ionic build microstructure may have specifically behavior.

The focusing observation both the ferrite material entropy and inversion degree from 0.35 to 0.45 will be studied in this article

II. Theoretical Basis.

The ferrite material characteristic are determined by many aspect such as; chemistry, crystal structure, and micro-structure which have extremely sensitive process. A long of every rod metal will obtain the different temperature at point to other point will give different free electron mobility.

2.1 The Seebeck Constant of The Cu-Zn Ferrite Material.

The ferrite cationic distribution is formulated as ;

$$(Me^{2+}_{(\delta)}Fe^{3+}_{(1-\delta)})\,[Me^{2+}_{(1-\delta)}Fe^{3+}_{(1+\delta)}]\,O_4 \quad [7] \quad \ldots\ldots 1)$$

Where Me are divalen metal such as; Zn^{2+},Cu^{2+}, Co^{2+},Fe^{2+} and etch.

The Crystalline structure of the Copper-Zinc Ferrite Spinel crystals consisting of 2 side tetra hedral crystals which side (side A) and the octahedral (B side) . Zn^{2+} cations occupy only tetrahedral side A. Zn Atom portion on the side of A determines the degree of inverse x . Cation Cu^{2+} and Fe^{2+} occupying octahedral side (side B) [8,9]

Based on ion concentration (y) divalent metal (Cu^{2+} , Fe^{2+}) and inverse parameter (x) formulation of cationic distribution CuZnFerrite written as equation ;

x as parameter of inversion degree .

y expressed consentrasi divalent Cu^{2+}

Thermoelectric properties of materials, mainly it has a Seebeck coefficient as [10]

$$S_{sebeck} = -\frac{k}{e}\left[Ln\left\{\frac{\beta.Fe^{3+}|oct}{Fe^{2+}|oct}\right\}\right] \ldots\ldots 3)$$

Where

Fe3 + | oct concentration of Fe^{3+} at the octahetral site

Fe2 + | Fe^{2+} concentration at the octahetral site

k (as Boltzman constant) = 8.6 x 10^{-5} eV β = 1, then constant of k/e=8.6x10^{-5} Volts.

The concentration of Fe^{2+} on octahedral sites linearly depends to the concentration of Zn, degree of inversion of the spinnel crystal is determined by the concentration of Zn , then if the inverse degree of x is increased Seebeck coefficient will be negative.

In practice it on arod metal, the Seebeck constant can be determined as equation

$$S_{seebeck} = \frac{\Delta V}{\Delta T} = \frac{[V_{T2} - V_{T1}]}{[T_2 - T_1]} \quad [8] \quad \ldots\ldots 4)$$

Where;

VT1 = Voltage electrical potential on the surface of the metal temperature in area 1.

VT2 = Voltage electrical potential on the surface of the metal temperature in area T2

T = absolute metal temperature.

A = dimensionless constant kinetic energy associated with the charge carriers.

at T = 300 K a = 0 at K = 800 K a = 2

Thermoelectric behavior is also obtained by the Ferrite semiconductor defect materials [10] but different with the metal conductor due to metal free electrons a, the free electrons of semiconductors occurs due to certain circumstances structure defect .

Thermoelectric properties appear of Cu substituted at Zn Ferrite at room temperature until the Curie temperature has been studied by DR.Ravinder in 2012 [9]

The Spinel materials having a similar degree of inverse AB2O4 x , the material configuration entropy is expressed by the equation 5 mainly the difference in configuration entropy between the magnetically-ordered and randomly-oriented spin states as equation

$$S_X = -R\{xLnx + (1-x)Ln(1-x) + xLn(x/2) + (2-x)Ln(1-x/2)\} \quad ......5) \quad [11]$$

Where

x as inversion degree.

R as the perfect gas constant

FIS method use co-precipitation process have main advantage that a large amount of nanoparticle can be synthesized but control of particle size is limeted due to only kinetik factor to control crystal growth. Mechanism of nucleation and groth following the LaMer diagram. .

Based on the principle of formation reaction with Ferrite the deposition method (co -precipitation) which requires the presence of Fe 2 + ion pair and Fe 3 + ions , while the Fe $^{2+}$ ions should be on the side of octahedral, then the distribution of cations Copper - Zinc Ferrite equation can be expressed as ;

$$(Me^{2+}_{(x)}Fe^{3+}_{(1-x)})[Me^{2+}_{(1-x)}Fe^{3+}_{(1+x)}]O_4 \text{ ,[6] ......6)}$$

Me are divalen metal such as; Zn^{2+},Cu^{2+}, Co^{2+},Fe^{2+} and etch.

Thepinel crystals consisting of 2 side tetra hedral crystals which side (side A) and the octahedral (B side) . Zn^{2+} cations occupy only tetrahedral side A. Zn Atom portion on the side of A determines the degree of inverse x . Cation Cu^{2+} and Fe^{2+} occupying octahedral side (side B) .

2.2 The Estimation Of Entropy Substance at The Flow Injction Method- FIS.

2.2.1 Calculation And Table Compilation Of

Avrami-Ozawa Parameter, Extracted From the data of pH Datalogger.

The Flow Injection Synthesis of co-precipitation process is done by means of the acid alkaline titration[12,]. Here the alkaline solution is pumped with a peristaltic pump and injected into an acid solution which is in the tub. During the titration process reactant solution was stirred with a mechanical stirrer at a speed of 500 RPM.

The table of Avrami-Ozawa parameter compilation specifically of process parameter at vary temperature, include the time of process, pH yield solution, rate of temperature exchange were extracted from datalogger[13]

Flow Injection Synthesis is one way to do co-precipitation synthesis which can be done automatically by using a data acquisition system supported datalogger instrument completed both high resolution of the digital pH meter and digital thermometer. In this study the data acquisition could be used to expected the necessary of performing material parameter such as the activation energy of crystallization, particle size, reaction rate, [8,14]

The thermodynamics of Cu-Zn ferrite behavior have been observed by many scientists since intensively in University of Michigan [8,16]] use sophisticated calorimeter. At room temperature is 5.367 [cal/mol] [8]. According to Cu-Zn ferrite as co-precipitation yield of chlorine Cu^{2+}, Zn^{2+}, $Fe^{3+}$and Fe^{3+} to alkaline hydroxide as precursor, the metal salts are in the negligible side, causing by suggest that all of input compound completely be amount of yields, but not in precipitant solution, the solution concentrate decrease proportional with increasing of yield concentrate [8,17], such as equation

$$\%Yield = \frac{\Delta Cpr_t}{\Delta Cpr_\infty} = \frac{Cpr_0 - Cpr_t}{Cpr_0 - Cpr_\infty} = \frac{10^{-pOH_0} - 10^{-pOH_t}}{10^{-pOH_0} - 10^{-pOH_\infty}} \quad \text{…………………………………………….} (8)$$

If the equation 1 as isothermal process, it can be involved with Avrami equation [16] such as

The entropy of substance-S can be calculated by devide the activation energy-ΔQ_E of average of the material forming reaction temperature use formulation as follow;

2.2.2 Execution of Rielveld Refinement

The Results of refinement by GSAS as shown in Figure 1, shows the residue and a very small calculation error. The Spinel structure formula was defined as AB_2O_4. Site A tetrahedral ontain (Cu and Zn) dan site B (Fe) octahedral located at Wyckoff positions, respectively 8(a) dand 16(d) with stoichiometric composition is defined as $[Cu^{2+}{}_{1-y-x}Zn^{2+}{}_{y}Fe^{3+}{}_{x}]^{A}[Fe^{3+}{}_{2-x}Fe^{2+}{}_{x}]^{B}O_4$ [8,17]

The pattern of X-ray diffraction (XRD) Rietveld refinement was continuously done until to get goodness factor value close to one. Below the Rielveld Refined for the typecal sampel $Cu_{(1-x)}Zn_xFe_2O_4$ wich the spacimnt code Bath04P, Bath08P are shown in figure 1

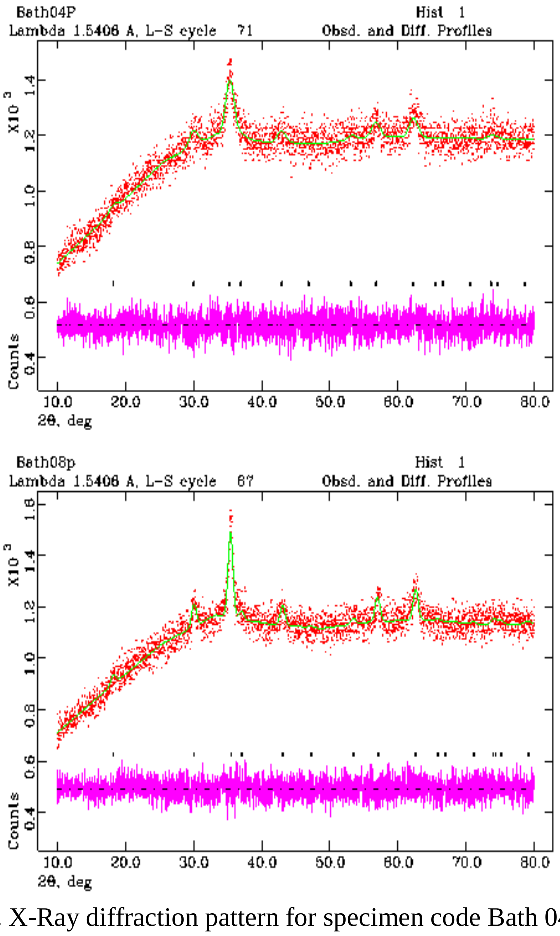

Fig.1. X-Ray diffraction pattern for specimen code Bath 04P, Ba

The results of the overall Bath 1,2,3 respectively till bath 9 is obtained as table 1.

Besides goodness factor, continues its will obtain the value discrepancy factor (R_{WP}) and expected (R_{exp}) with goodness index χ^2 as listed in Table 1.

The speciment code contains dissection on the contents of the tetrahedral lattice and lattice octahedral. The tetra hedral containing Cu^{2+}, Zn^{2+} and Fe^{3+} while octahedral containing Fe^{2+} and Fe^{3+} , Containing Fe^{2+} in the octahedral must the same with containing Fe^{3+} in tetrahedral lattice. Actual content both is called inversion degree. Based on the equation 5 is related to the entropy S.

The Inversion degree of the speciment code bath08P have 0.4516 and coeffisien seebeck -10.84 [10^{-2}mV] , as a relatively privileged relationship. a symbol of equalization. From this equalization begins

Table 1. The list parameter value of R_{wp},R_{exp} and χ^2

Specimen Code	R_{wp}	R_{exp}	χ^2	Lattice constant a [^{0}A]	(A) Tetrahedral			[B] Octahedral		InversionDegree	Chemical Formula Yield
					Cu^{2+}	Zn^{2+}	Fe^{3+}	Fe^{2+}	Fe^{3+}		
Bath04P	3.10	2.47	1.096	8.441(1)	**0.4951**	**0.0828**	**0.3867**	**0.3867**	**1.6485**	**0.3867**	$Cu_{0.4951}Zn_{0.0828}Fe_{2.4220}O_4$
Bath06P	3.14	2.52	1.090	8.423(9)	**0.5382**	**0.1283**	**0.3647**	**0.3647**	**1.6040**	**0.3647**	$Cu_{0.5382}Zn_{0.1283}Fe_{2.3335}O_4$
Bath08P	3.13	2.5	1.072	8.390(5)	**0.4862**	**0.0199**	**0.4516**	**0.4516**	**1.5907**	**0.4516**	$Cu_{0.4862}Zn_{0.0199}Fe_{2.4939}O_4$
Bath085P	3.15	2.51	1.115	8.390(6)	**0.4468**	**0.2221**	**0.3900**	**0.3900**	**1.5511**	**0.39**	$Cu_{0.4468}Zn_{0.2221}Fe_{2.3311}O_4$
Bath088P	3.12	2.49	1.075	8.407(6)	**0.4798**	**0.1414**	**0.3938**	**0.3938**	**1.5912**	**0.398**	$Cu_{0.4798}Zn_{0.1414}Fe_{2.3788}O_4$
Bath-09P	3.04	2.47	1.048	8.470(1)	**0.4636**	**0.0215**	**0.3963**	**0.3963**	**1.7222**	**0.3963**	$Cu_{0.4636}Zn_{0.0215}Fe_{2.5149}O_4$
Bath-0 P	3.16	2.52	1.094	8.507(2)	**0.4292**		**0.3030**	**0.3030**	**1.9648**	**0.3030**	$Cu_{0.4292}Fe_{2.5708}O_4$

Cation distribution separately tetrahedral and octahedral automatically separately lattice volume, inversion degree and constan coeffisien like in table

Table 2, Cation Distribution in speciment code, results lattice constanta, lattice volume and Seebeck Coefficient

Speciment Code	**cationic Distributions**					Lattice constanta [^{0}A]	Lattice Volume	Inversion Degree	Entropy	Seebeck Coeff _α [10^{-2}mV]
	Tetrahedral			**Octahedral**						
	Cu^{2+}	**Zn^{2+}**	**Fe^{3+}**	**Fe^{2+}**	**Fe^{3+}**	**A=b=c**	**V [A^3]**	**x**	**S**	**□=(k/e) Ln{Fe^{2+}/Fe^{3+}}**
Bath 04P	**0.4951**	**0.0828**	**0.3867**	**0.3867**	**1.6485**	8.441(1)	**601.5**	**0.387**	3.215411	**-12.47**
Bath 06P	**0.5382**	**0.1283**	**0.3647**	**0.3647**	**1.6040**	8.423(9)	**597.6**	**0.3647**	3.032481	**-12.73**
Bath 08P	**0.4862**	**0.0199**	**0.4516**	**0.4516**	**1.5907**	8.390(5)	**590.7**	**0.4516**	3.755054	**-10.84**
Bath 085P	**0.4468**	**0.2221**	**0.3900**	**0.3900**	**1.5511**	8.390(6)	**590.7**	**0.3900**	3.24285	**-11.78**
Bath 088P	**0.4798**	**0.1414**	**0.3938**	**0.3938**	**1.5912**	8.407(6)	**594.2**	**0.3938**	3.274447	**-11.95**
Bath 09P	**0.4636**	**0.0215**	**0.3963**	**0.3963**	**1.7222**	8.470(1)	**607.6**	**0.3963**	3.295235	**-12.56**
Fisbath	**0.4292**		**0.3030**	**0.3030**	**1.9648**			**0.3867**		

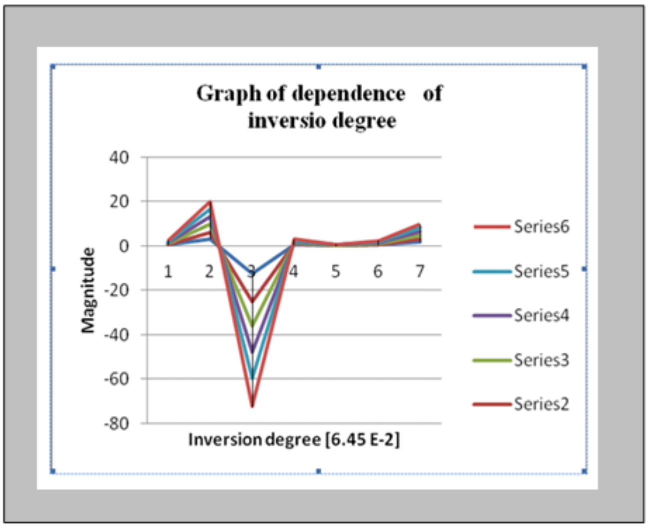

Fig.2 The graph of the dependency cu-Znferit behavior of the Cu-Znferrite inversion degree.

In the all of series graph the inversion degree give strongly influent but at the 2.3x6.45E-2, 4x6.45E-2 and 6x6.45E-2 point respectively the inversion degree almost did not give dependency to the ferrite behavior..

III. Discussion

At the figure 3, there are 6 lines. Each line show of speciment code characteristic, such as example line 6, the speciment code is Bath09.In this node the entropi is 3.29 relatively low.

The ferrite material have lot of parameters may be observation object to explore behavior of subtance, but only seven parameters will be observed include ineversion degree parameter as the centre case [18]. The parameters i.e, 1. fraction of both cation irons Fe^{3+} and Fe^{2} in the octahedral site, fraction of divalent metal cations in tetrahedral site i.e Cu^{2+}, and Zn^{2+}, entropi of ferritesubstance , and Seebeckconstant. The sevent parameters sstisfy to estimate of Seebeck constant anf thermoelectric behavior of Cu-Zn Ferrite. Ferrite material which is relatively unstable will have the higher entropy. Given the algebraic entropy is a logarithmic function of the degree of inversion of the degree of entropy will be able to have several peaks exponents modulations.

To ensure that ferite will have a low degree of inversion can be approached by use stoichiometry that divalent metal fraction of the three-valent iros (Me^{2+} / Fe^{3+})> 0.5.

As a result the graph of the figure 3 obviously shows that the degree of inversion has a strong role to determining properties of the ferrite material which in a relatively narrow interval nodes of inversion i.e from 0.35 to 0:45 there are 7-six stables and 4-four unstable state where the stability of state is expressed by the value of entropy- S

In the Table 3 shows that the degree of inversion is below 0.5 means the use of Fe^{2+}-based compounds with the formula (Fe^{2+}+Me^{2+})/ Fe^{3+} =<0.5 will result the inverse spinel with an inversion less than 0.5. If the doping atoms are Cu and Zn it will generate a lot of distortion.

The figure 3, the graph of degree inversion x versus Zn cationic fraction showed linearity ramps due by the radius of Zn^{2+} is greater then other cationic and prove that the degree of inversion of the spinel Cu-ZnFerrite beside be affected by Zn^{2+} but also another cationic such as Cu^{2+} and Fe^{3+}.

In the table 3, shows the list of the cationic both di-valences and tree valences include inversion degree a enthalpy and entropy have been estimated by Temkin formula.. For spinnel have degree inversions 0.303 until 0.4516 the entropy value of 2.52 to 3.76

The tetrahedral sites are contained by both cationic of Cu^{2+}, Zn^{2+} and Fe^{2+}. The volume of its beside are depends by fraction of tetrahedral ionic site but also fraction of octahedral site contain cationics of Fe^{2+} and Fe^{3+} then the tetrahedral sides volume greater than octahedral.
The comparative of cationic radius such as; $Fe^{2+} > Zn^{2+} > Cu^{2+} > Fe^{3+}$.

The Cu2+ cations are in a tetrahedral lattice is not due to a relatively small but the Cu^{2+} cations has a high reactivity to merge with ionic ligand form a tetrahedral structure.

At the same figure 3 see the Cu2+ cationics give an effect significantly to decreas the tetrahedral volume, the tetrahedral size is dominated by Cu^{2+} ion size due to the amount of Cu fraction more than the fraction of the cationic Zn^{2+}.

Increasing of the Cu^{2+} contain cause sharp decreasing of the Spinnel volume, indicate tetrahedral volume very sensitive to spinnel CuZnFerrite volume decrase. This decreasing will cause the different energy level.

The dependence of both the Seebeck constant entropy and inversion parameters to reviewing requires precise fitting and thoroughly tendentious, a strong trend towards the parameters are not necessarily but it may representative of other parameters. The entropy substance relating to magnetism material is a parameter that can be measured both visually and by instrument.

The CuZnFerrite tend to irregular the entropy tend to higer by existing of Cu^{2+} and Fe^{2+} due increasing of inversion degree. The decreasing of tetrahedral volume due to increasing of Cu^{2+} will give volume unbalance between tetrahedral and octahedral fig. The condition will have an impact include; instability of chemical composition, instability due to the electron migration of $Fe^{2+}$to octahedral site due to exchange of Cu^{2+} to Cu^{1+}.

IV. Summary

Using the chart at fig.3 shows the node 3 have potential information that ferrite in very unstable state is in line series 3. The entropy ferrite substance value is 3.76 [J/mol K] and the optimum Seebeck constan is -10.84[E-2mV/K]= -108.4 [uV/K] almost the same with result of Dawoud [11] i.e in a hundred around uV/K and behaves as n-type semiconductor. The fig.2 the line number 6 (orange line) and line number 5 (show three valent irons dominate then all of parameters, it means the cationic of Fe^{3+} and Fe^{2+} especially in the nodes 3 is in unstable state cause the electron migration from unstable cationics to relatively cationic stable, expected cationics Cu and Fe as the causedd.

Reaction formation cuznferrite material deposition method performed in flow injection synthesis reactor can be done, but it requires the agglomeration prevention materials. Mean also allows to determine the In the Table 1 shows that the degree of inversion is below 0.5 means the use of Fe 2 +-based compounds with the formula Fe 2 + / Fe 3 + = 0.5 will result in an inverse spinel with an inversion below 0.5. If the doping atoms are Cu and Zn it will generate a lot of distortion.

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