The Effect of Sintering Atmosphere on Electrical Characteristics of Fe$_2$TiO$_5$ Pellet Ceramics Sintered at 1200°C for NTC Thermistor

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Abstract. Fabrication of Fe$_2$TiO$_5$ pellet ceramics using powder metallurgy technique for NTC thermistor has been carried out, in order to know the effect of sintering atmosphere (Oxygen, Air and Argon Gas) on the characteristic especially the electrical characteristic of Fe$_2$TiO$_5$ ceramics with high working temperature. X-ray diffraction analyses (XRD) was done to know crystal structure and phases formation. A SEM analysis was carried out to know microstructure of pellets. Electrical properties characterization was done through measurement of electrical resistance at various temperatures (room temperature to 250°C). The XRD data showed that the pellets crystallize in orthorhombic. The presence of second phase could not be identified from the XRD analyses. The SEM images showed that the grains size of the ceramic sintered in oxygen gas is smaller than that of the ceramic sintered in air and argon gas. Electrical data showed that the pellet ceramics sintered in oxygen gas had the largest room temperature resistance ($R_{RT}$), thermistor constant ($B$), activation energy ($E_a$) and sensitivity ($\alpha$) compared to those sintered in air and argon gas. From the electrical characteristics data, it was known that the electrical characteristics of the Fe$_2$TiO$_5$ pellet ceramics followed the NTC characteristic. The value of $B$ and $R_{RT}$ of the produced Fe$_2$TiO$_5$ ceramics namely $B = 4389-6149$ K and $R_{RT} = 342-26548\Omega$, fitted market requirement and can be used for temperature sensor.

1. Introduction

Negative Temperature Coefficient (NTC) thermistor has been widely used around the world today, due to its capability used in various fields of electronics, such as thermometer, electric current limiter, water flow sensor, and pressure sensor [1-2]. The NTC thermistor is generally made of ceramic having structure of spinel of AB$_2$O$_4$ where A is the ion occupies tetrahedral position and B is the ion occupies octahedral position [3]. Fe$_2$TiO$_5$ ceramic is one of some ceramics that can be applied for NTC thermistor. The thermistor may be produced in the form of disk/pellet or thick film. Here, the object of study is the pellet thermistor.

In this work, a study on fabrication of pellet thermistor based on Fe$_2$TiO$_5$ with sintering atmosphere variation was performed. The effect of sintering atmosphere on the characteristics, especially the electrical characteristics, of the Fe$_2$TiO$_5$ pellet ceramics for NTC thermistor was discussed. Fe$_2$TiO$_5$ is one of semiconductor ceramics used as based material for main components fabrication of NTC thermistor as temperature sensor. The composition of mineral Fe$_2$TiO$_5$ is belong to pseudobrokyte group where the general formula of this compound is X$_2$YO$_5$ with octahedral in both side, X and Y. Generally, Fe$_2$TiO$_5$ has been mostly used for gas sensor, non linear optic, magnetic, catalyst, and microelectronics [4]. Since Fe$_2$TiO$_5$ actually has semi-conductivity, it is very capable to use Fe$_2$TiO$_5$ for NTC thermistor as based material. Thermistor constant $B$ is a quantity which determine typical characteristic of thermistor corresponding to electrical resistance changes with temperature. The larger...
thermistor constant lead to better thermistor quality. Many works and studies have been conducted by researchers to enhance thermistor constant $B$ and thermistor sensitivity $\alpha$. They have studied the effect of sintering atmosphere to typical characteristic of Fe$_2$TiO$_5$-based ceramics. The research of NTC thermistor for high temperature has been previously reported [5]. However, the reports of NTC thermistor in high temperature fabricated by Fe$_2$TiO$_5$-based ceramics have not been excessively reported so far.

2. Material and Method

Fe$_2$TiO$_5$ thermistor ceramic was prepared by using imported Fe$_2$O$_3$ and TiO$_2$. Mixture of Fe$_2$O$_3$ and TiO$_2$ with each compound ratio 50:50 in % mole, was calcinated at 700°C for 2 hours. In order to form pellets, pressed powder, the homogeneous mixture of Fe$_2$O$_3$ and TiO$_2$ was pressed at 4.10$^3$ kg/cm$^2$. The pellets were sintered at 1200°C for 2 hours in oxygen gas, air and argon gas. In order to observe crystal structure and formed phases, Fe$_2$TiO$_5$ pellets were analyzed by x-ray diffraction (XRD) with Kα radiation at 40 kV in voltage and 25 mA in current. Microstructure photographs of the Fe$_2$TiO$_5$ sintered pellets were carried out by an electron microscope (SEM). Electrical characteristic was carried out by measuring electrical resistance at various temperatures up to 250°C by 10°C interval. Both surfaces of sintered pellets had been coated by conductive silver paste colloid silver solution and heated at 600°C for 10 minutes in advance. Thermistor constant ($B$) was derived from Ln resistivity vs. $1/T$ curve where $B$ is the gradient of the curve based on [6-8]:

$$R = R_0 e^{\frac{B}{T}}$$

Where, $R$ is the electrical resistance, $R_0$ is a constant or the resistant at the infinite temperature, $B$ is the thermistor constant and $T$ is the temperature in Kelvin and $k$ is the Boltzmann constant. Room temperature resistance ($R_{RT}$) was determined as the electrical resistance at room temperature (25°C). From the value of $B$, the activation energy ($E_a$) and sensitivity ($\alpha$) were calculated using equation (2) and 3 [9-12].

$$E_a = Bk$$

$$\alpha = \frac{B}{T_2}$$

3. Result and Discussion

XRD profiles of Fe$_2$TiO$_5$ pellet ceramics sintered at 1200°C for 2 hours with sintering atmosphere of oxygen (sample A), air (sample B), and argon gas (sample C) respectively are shown in Figure 1, Figure 2 and Figure 3. As shown in the Figure 1, Figure 2 and Figure 3 the profiles are generally similar. The XRD profiles show that the structure of the pellet ceramics is orthorhombic after being compared to the XRD standard profile of Fe$_2$TiO$_5$ from JCPDS No. 01-070-2728). No peaks from second phases observed. It may be due to the small concentration of impurities which is smaller than the precision limit of the x-ray diffractometer used. The XRD data of Figure1-Figure 3 indicates that the synthesis of the Fe$_2$TiO$_5$ pellets has been well prepared from Fe$_2$O$_3$ and TiO$_2$ powder with sintering atmosphere condition.
Figure 1. XRD profile of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample A.

Figure 2. XRD profile of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample B.

Figure 3. XRD profile of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample C.
Microstructures of the Fe$_2$TiO$_5$ pellet ceramic sintered at 1200°C for 2 hours in sample A, sample B, and sample C respectively, are showed in Figure 4, Figure 5 and Figure 6. All of the pellets are characterized in porous structure with different grain size depending on the sintering atmosphere condition. The samples could be well synthesized with oxygen, air and argon gas sintering atmospheres. The SEM images showed that the pellet ceramics sintered in oxygen gas had the smallest grain size, compared to those sintered in air and argon gas. This situation can be explained as follow a relatively poor oxygen for sample sintered in argon gas, make the ceramic could not be well synthesized. Some of Fe$_2$O$_3$ and TiO$_2$ segregated at grain boundaries and inhibited grain growth, producing small grains. The grain size calculated by using of the intercept method is found to be 0.510μm, 0.604μm and 1.127μm for sintered in sample A, sample B, and sample C, respectively.

Figure 4. Microstructure of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample A.

Figure 5. Microstructure of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample B

Figure 6. Microstructure of Fe$_2$TiO$_5$ based-pellet sintered at 1200°C for sample C
The electrical data of the Fe₂TiO₅ pellet ceramics with sintering atmosphere variation is shown in Figure 6 and Table 1. The electrical data of Figure 6 shows that the Ln resistivity increases linearly as the 1/T increases, indicating that the electrical characteristics of the ceramics follows the NTC tendency expressed by equation (1). As shown in Table 1, electricity resistance and thermistor constant of Fe₂TiO₅ based-pellet sintered at 1200°C for 2 hours in sample A is larger than those of Fe₂TiO₅ based-pellet fired at 1200°C for 2 hours in sample B and sample C. The ceramics sintered in sample A have relatively smaller oxygen vacancy and means smaller number of electron, so the resistance of this ceramic is larger. The grains of the ceramic sintered in sample A is smaller than that of the ceramic sintered in sample B and sample C, the resistance of this ceramic is lower than that of the ceramic sintered in other atmospheres. This is due to the larger number of oxygen vacancy, that means larger number of electron, contained in the ceramic sintered in argon gas. The value of thermistor constant (B) ceramics fitted market requirement for NTC thermistor.

![Figure 7](image-url)  
**Figure 7.** The relation between Ln Electrical Resistance and 1/T of Fe₂TiO₅ based-pellet sintered at 1200°C for sample A, sample B, and sample C.

| Sample | Sintering atmosphere | Thermistor constant (B) (K) | Activation Energy (Eₘ) (eV) | Sensitivity (α) (% K⁻¹) | Room temperature resistance (R_RT) (kΩ) |
|--------|----------------------|-----------------------------|-----------------------------|-------------------------|----------------------------------------|
| A      | Oxygen               | 6149                        | 0.530                       | 6.9                     | 26548                                  |
| B      | Air                  | 5879                        | 0.507                       | 6.6                     | 13201                                  |
| C      | Argon                | 4389                        | 0.378                       | 4.9                     | 342                                    |
4. Conclusion

Fe$_2$TiO$_5$ pellet ceramics have been well sintered at 1200°C for 2 hours with three different atmospheres i.e. oxygen, air and argon gas. All of the pellets crystallize in orthorhombic structure. The pellet ceramics sintered in oxygen gas have largest resistance, thermistor constant, activation energy and sensitivity compared to those sintered in air and argon gas. The electrical characteristics of the Fe$_2$TiO$_5$ pellet ceramics follow the NTC characteristic. The value of thermistor constant ($B$) = 4389-6149 K and room temperature resistance ($R_{RT}$) = 342-26548 kΩ of the produced Fe$_2$TiO$_5$ ceramics fit market requirement and can be used for temperature sensor.

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