

## Decomposition of Zn-ferrite for O<sub>2</sub> generation by concentrated solar radiation

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### Abstract

The O<sub>2</sub> generation step in two-step water splitting with Zn-ferrite/Zn, Fe<sub>3</sub>O<sub>4</sub> system was studied with a stainless steel reactor having a window of quartz glass plate (Sandwich Reactor) using the 15 kW solar dish concentrator in Australian National University. The reactant flat layer with a small thickness in the Sandwich Reactor was assumed to simulate the flow section of the particle cloud reactor. The temperature of the sample attained 1750 K in a few minutes by concentrated solar radiation, and 40% molar of the Zn-ferrite was decomposed into Fe<sub>3</sub>O<sub>4</sub> to produce Zn<sub>0.4</sub>Fe<sub>2.6</sub>O<sub>4</sub>, which is the solid solution between ZnFe<sub>2</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>. The laboratory experiment with the Xe beam irradiation showed that the Zn-ferrite decomposition reaction proceeds according to  $\frac{3}{2}\text{ZnFe}_2\text{O}_4 = \frac{3}{2}\text{Zn} + \text{Fe}_3\text{O}_4 + \text{O}_2$ . In the solar furnace experiment, the metal Zn vapor produced according to this equation seems to be deposited on the surface of the inner wall of the reactor, or it seems to recombine with the released O<sub>2</sub>, converting to ZnO under the condition without quenching system in the present experimental setup.

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### 1. Introduction

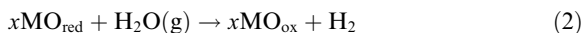
Many approaches for utilizing solar energy have the disadvantage that they do not offer means for storing and transporting the energy. Utilizing solar energy for the production of solar fuels (Steinfeld, 1993; Tamaura et al., 1995, 1997) solves this problem. Solar fuels can be stored until required and transported to any consuming site. A promising solar fuel form is methanol (or DME, dimethyl ether). Methanol (or DME) can be regarded as another form of hydrogen (H<sub>2</sub> is combined with the carbon and can be stored and transported in the liquid form, which can be more easily handled than gaseous H<sub>2</sub>). Methods to produce H<sub>2</sub> from solar energy have been

explored widely. One of the two-step water-splitting processes uses metal oxides (MO<sub>red</sub>/MO<sub>ox</sub>) as a redox pair (Kogan et al., 2000; Getoff, 1990; Nakamura, 1977; Steinfeld et al., 1998; Tamaura et al., 1999; Hosokawa et al., 1999). The reaction system can be written as:

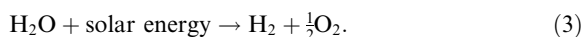
1st (O<sub>2</sub> generation (also energy storage)) step:



2nd (H<sub>2</sub> generation) step:



where MO<sub>red</sub> and MO<sub>ox</sub> denote the reduced and oxidized states respectively. The net reaction is:



In the 1st step, metal oxide is transformed to the reduced forms (MO<sub>red</sub>) at high temperatures, where  $\Delta G^0$  is negative for the O<sub>2</sub> generating direction of the reactions.

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In the 2nd step,  $H_2$  is generated by the reaction of  $MO_{red}$  with  $H_2O$ .

Iron oxides and their related compounds are attractive candidates for this kind of system, as they have a variety of oxides with different oxidation states. There are a number of possible combinations. Carbon bearing magnetite and  $NiFe_2O_4$  can be activated at low temperature of 573 K (Akanuma et al., 1993; Tamaura et al., 1994; Sano et al., 1996a,b). However, in this system, the amount of hydrogen generated in the reaction is very small, since the water splitting system is based on the non-stoichiometry of ferrite between ferrite and activated ferrite (cation-excess ferrite) (Tamaura et al., 1998). In (Ni,Mn)-ferrite system, the amount of hydrogen generated in the water splitting reaction is also limited (0.02 mol of  $H_2$  is generated from 1 mol of ferrite), since the water splitting utilizes the redox system with non-stoichiometric ferrite (Kojima et al., 1996). To obtain a larger amount of hydrogen, the two-step water splitting system with Mn-ferrite ( $MnFe_2O_4/Na_2CO_3$ ) has been studied (Ueda et al., 1998; Tamaura et al., 1999). A new three-step water splitting with  $MnFe_2O_4/Na_2CO_3/Fe_2O_3$  system has also been proposed (Hosokawa et al., 1999; Kaneko et al., 2001), and more than 0.5 mol of  $H_2$  is generated from 1 mol of ferrite with this system. In previous investigations, a mixture of Zn and magnetite ( $Fe_3O_4$ ), heated to 873 K with steam passed through, gave the 2 mol of  $H_2$  from 1 mol of magnetite with the formation of 1.5 mol Zn-ferrite (Tamaura et al., 2001). Zn-ferrite ( $ZnFe_2O_4$ ) can be reduced by thermochemical decomposition at high temperature to generate Zn,  $Fe_3O_4$  and  $O_2$ . It has been found that small  $\Delta G^0$  for the 1 mole  $O_2$  generation reaction is obtained in Zn-ferrite above 1500 K through thermodynamic calculations (Kaneko et al., 2002).

We the two-step water splitting system with the Zn-ferrite/Zn,  $Fe_3O_4$ , which can be used for the solar/chemical energy conversion process. The schematic outline of this system is shown in Fig. 1. This system consists of two steps:  $O_2$  generation and  $H_2$  generation step. In the first ( $O_2$  generation) step, Zn-ferrite reacts releasing Zn and forming  $Fe_3O_4$  above 1500 K in an Ar flow obtained by irradiating with a concentrated solar beam. In the second step, the mixture of  $Fe_3O_4$  and Zn reacts with water vapor to generate hydrogen and forms  $ZnFe_2O_4$  at 873 K. Concentrated solar radiation can easily produce temperatures above 1500 K. Direct irradiation of such high flux solar radiation may provide effective decomposition of Zn-ferrite with  $O_2$  generation and solar thermal production of  $H_2$  from  $H_2O$  with the Zn-ferrite/Zn,  $Fe_3O_4$  system. In this paper, an investigation of the decomposition reaction of Zn-ferrite for  $O_2$  generation by concentrated solar radiation is reported. A unique low cost solar reactor was designed in order to examine the feasibility of performing the thermal decomposition of Zn-ferrite with concentrated solar radi-

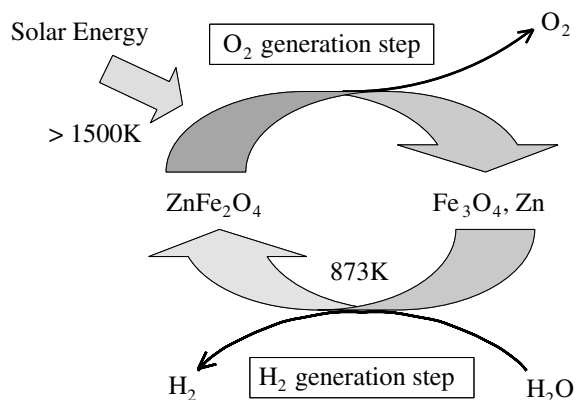


Fig. 1. The schematic outline of two-step water splitting system with Zn-ferrite/Zn,  $Fe_3O_4$  system.

ation. The decomposition ratio of Zn-ferrite with various temperatures has been investigated via solid product analysis.

## 2. Experiments with a low cost “sandwich reactor”

In order to test the feasibility of carrying out the thermal decomposition of Zn-ferrite with concentrated solar radiation, a unique low cost solar reactor design was devised and tested on the Australian National University’s 20 m<sup>2</sup> paraboloidal dish solar concentrator.

The solar reactor concept has been dubbed the “sandwich reactor” reflecting its method of construction, which is illustrated in Fig. 2. It is a simple batch reactor designed for short term transient operation. In operation a sample of the reactant is positioned against a flat quartz window on a bed of quartz wool which is 20 mm deep. The wool is held inside a steel ring that sits in

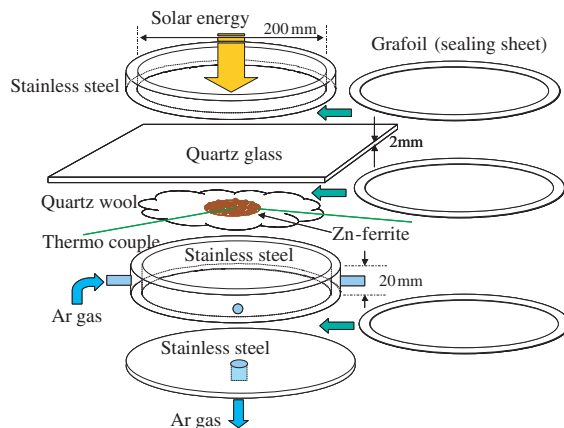


Fig. 2. Experimental apparatus for the  $O_2$  generation reaction with Zn-ferrite system.

between the quartz window and a steel backing plate. The whole arrangement is held together with simple screw clamps, with “Graphoil” sealing gaskets between the various components. Provision is made for circulation of Ar gas through the reaction chamber at low pressure.

Calculations of the transient response in temperatures expected when the reactor faces the concentrated solar radiation, indicated that with the materials chosen, the Zn-ferrite sample would reach reaction temperatures in a few seconds, whereas the vulnerable components of the reactor would take several minutes before they would reach temperatures that could damage them.

It was found that the design did function successfully, however in initial tests, the quartz window, where it was in contact with the Ferrite powder, exceeded its melting point and the small internal pressure was sufficient to blow it out. This was rectified by adding a second smaller piece of quartz inside the main window and spaced 2 mm from it with packing in its corners. In effect constructing a “double glazed” window, the smaller piece of quartz in direct contact with the Ferrite powder experienced the highest temperatures, but because it was not exposed to any pressure difference, it was not forced out of shape.

Fig. 3 shows the reactor mounted on the ANU dish. The small “sandwich reactor” was mounted inside the aperture of a cavity receiver/reactor which had been previously installed. The larger receiver/reactor played no part in the experiment, it simply provided a convenient means for mounting the new reactor.

In the experiments conducted, the Zn-ferrite powder was supplied by Toda Kogyo Corp. Two Pt–Pt/Rh thermocouple sensors were used to measure the temperature of the reactant powder just behind the window surface. An Ar flow of 1000 cm<sup>3</sup>/min was maintained.

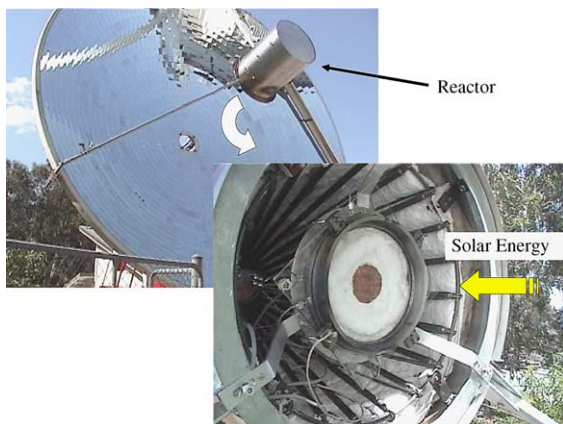


Fig. 3. 15 kW solar furnace using the O<sub>2</sub> generation reaction (ANU in Australia) and the “sandwich reactor” mounted inside.

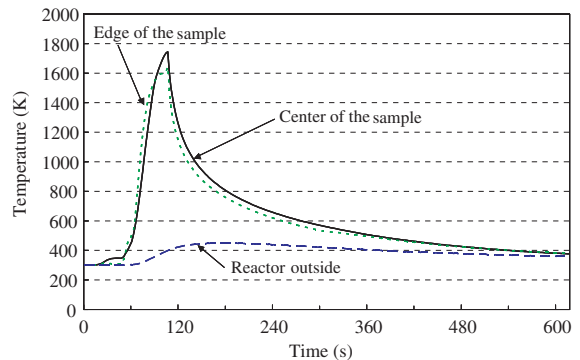


Fig. 4. Temperature change of sample and reactor by the solar furnace.

Fig. 4 illustrates a typical response of reactor temperatures when the dish and reactor was rapidly aligned with the sun. The period over which the reactor was on sun was just over 60 s. It took less than 40 s for the Zn-Ferrite to reach an expected reaction temperature of 1500 K. After a further 20 s the sample had reached 1750 K after which the dish was taken off the sun. The insolation level was 840–980 W/m<sup>2</sup> during this period. During this time the temperature of the outer structure of the reactor only rose to 450 K.

### 3. Analysis of reaction products

The solid phase was identified by X-ray diffractometry (XRD) (RIGAKU, RINT 2100) with FeK $\alpha$  radiation (30 kV, 20 mA). The lattice constant  $a_0$  of the product was calculated by extrapolating the values of  $a_0$  vs. the Nelson–Relay function,  $\cos^2 \theta / \sin \theta + \cos^2 \theta / \theta$ , to zero using the least-squares method (Nelson and Riley, 1945; Taylor and Sinclair, 1945; Geiss, 1961). In addition, Mössbauer spectra were measured with a <sup>57</sup>Co source diffused in metallic Rh. The spectra were calibrated with an iron foil absorber and were analyzed by computer fitting to a Lorentzian curve using a non-linear square routine. The Fe<sup>2+</sup>/Fe<sub>total</sub> mole ratio of the sample was determined by colorimetry (Iwasaki et al., 1960) using 2,2'-bipyridil (Hitachi Spectrometer, Model 124). The amount of iron and zinc in the product was measured by atomic absorption spectroscopy (Shimadzu, AA-6650F) after dissolving the sample in a hydrochloric acid solution.

Fig. 5 shows the XRD pattern for the solid product of the solar decomposition reaction of the Zn-ferrite at 1773 K. The XRD pattern showed a spinel type structure the same as the pattern for the Zn-ferrite, and peaks corresponding to ZnO and Zn were observed. The lattice constant  $a_0$  for the solid product with spinel type structure was 0.8424 nm, which is smaller than 0.8441 nm for

the Zn-ferrite and close to 0.8397 nm for the  $\text{Fe}_3\text{O}_4$ . This result shows that the product solid is the spinel type compound given by  $\text{Zn}_x\text{Fe}_{3-x}\text{O}_4$ , and that the Zn-ferrite was reduced by solar thermochemical decomposition with generation of  $\text{Zn}(\text{g})$ ,  $\text{Zn}_x\text{Fe}_{3-x}\text{O}_4(\text{s})$  and  $\text{O}_2(\text{g})$ . Fig. 6 shows the relationship between the lattice constant of the solid products obtained by the solar decomposition reaction of the Zn-ferrite and the reaction temperature. It can be seen that the solar decomposition of the Zn-ferrite commenced above 1500 K and its decomposition ratio at 1750 K was evaluated to be 40% from the lattice constant measurement. It is apparent that the solar decomposition reaction of Zn-ferrite can be readily promoted with increasing temperature.

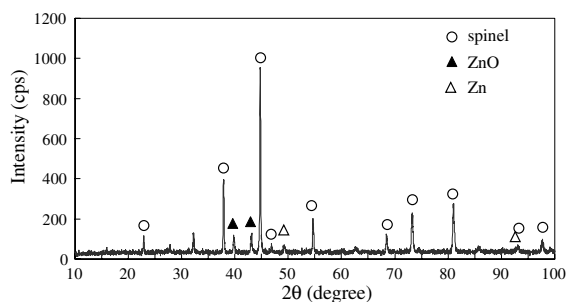


Fig. 5. XRD pattern for solid product after the Zn-ferrite (solar thermal) decomposition reaction at 1750 K.

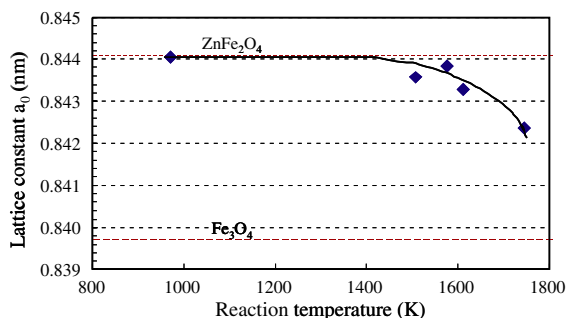


Fig. 6. Lattice constant  $a_0$  of the solid products at various decomposition temperatures.

The relationship between the mole ratio of  $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$  of the solid products obtained by the solar decomposition of the Zn-ferrite and the reaction temperature is given by Fig. 7. The  $\text{Fe}^{2+}$  contents in the solid product increased with an increase in the reaction temperature. Chemical analysis showed that about 40% moles of the  $\text{Fe}^{3+}$  ions in the Zn-ferrite were reduced to  $\text{Fe}^{2+}$  at 1750 K, and that this reduction reaction proceeds above 1500 K. The decomposition ratios of the Zn-ferrite evaluated from the mole ratio of  $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$  in the solid product were coincident with those lattice constant measurements. These data concerning the relationship between the lattice constant and the mole ratio of  $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$  are listed in Table 1. The lattice constant of the solid spinel product was decreased with an increase in the  $\text{Fe}^{2+}$  content in the solid product. About 13% moles out of the total iron ions in the solid product were in the form of  $\text{Fe}^{2+}$ , indicating that 40% moles of the Zn-ferrite is converted to  $\text{Fe}_3\text{O}_4$  at 1750 K by the concentrated solar radiation. This reduction coincides with the decrease in the lattice constant of the solid solution between  $\text{ZnFe}_2\text{O}_4$  and  $\text{Fe}_3\text{O}_4$  ( $\text{Zn}_x\text{Fe}_{3-x}\text{O}_4$ ) ( $x = 0.04\text{--}0.4$ ). And the lattice constant of the solid product reduced by 40% was almost equal to that of the solid solution of  $\text{Zn}_{0.4}\text{Fe}_{2.6}\text{O}_4$ .

Those findings for the reduction of the Zn-ferrite (decomposition reaction) by the concentrated solar radiation suggests that there two reactions are occurring given by

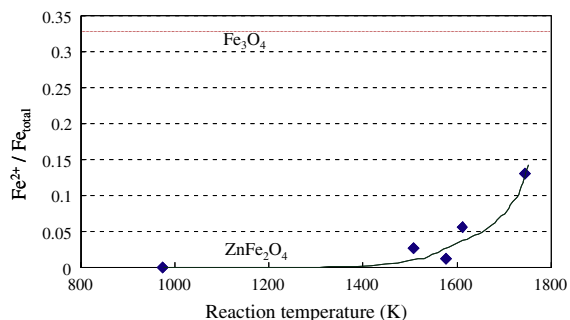
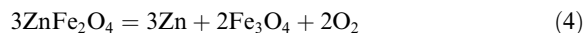


Fig. 7. Mole ratio of  $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$  of the solid products at various decomposition temperatures.

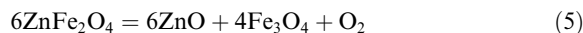
Table 1

Lattice constant ( $a_0$ ), contents of  $\text{Fe}^{2+}$  and mole ratio of Zn/Fe in solid products

	Lattice constant $a_0$ (decomposition ratio)	Mole ratio of $\text{Fe}^{2+}/\text{Fe}_{\text{total}}$ (decomposition ratio)	Mole ratio of Zn/Fe
$\text{ZnFe}_2\text{O}_4$	0.8441 nm	0.000	0.5
$\text{O}_2$ generation at 1510 K	0.8436 nm (7.9%)	0.012 (3.6%)	0.552
$\text{O}_2$ generation at 1610 K	0.8433 nm (20%)	0.056 (17%)	0.470
$\text{O}_2$ generation at 1750 K	0.8424 nm (41%)	0.130 (39%)	0.491
$\text{Fe}_3\text{O}_4$	0.8397 nm	0.333	(0.0)



and



From the laboratory experiment using the Xe beam irradiation for the decomposition reaction of Zn-ferrite at high temperatures ( $>1273$  K), the oxygen evolution was confirmed by the MS measurement of the product gas. Also, the deposition of the metal Zn on the cold finger for quenching was observed (Kaneko et al., 2002). Thus, it can be concluded that the Zn-ferrite decomposition reaction by the irradiation of the concentrated beam mainly proceeds with the release of  $\text{O}_2$  according to Eq. (4). However, as can be seen from Table 1, the decreasing of the Zn content in the solid product obtained in the solar reactor was not observed with decomposition of the Zn-ferrite. In the solar experiment, the metal Zn vapour produced according to Eq. (4) was presumably deposited on the surface of the Zn-ferrite particles in the reactor, or recombined with the released  $\text{O}_2$  converting to ZnO (in the absence of a quenching system in the present experimental setup).

The Mössbauer study of oxygen-deficient Zn-ferrites ( $\text{Zn}_x\text{Fe}_{3-x}\text{O}_{4-\delta}$ ,  $0 \leq x \leq 1$ ) was reported previously (Tabata et al., 1994). The Mössbauer spectra of the Zn-

ferrite (starting material) and the solid product after the solar experiment are shown in Fig. 8. In the spectrum of the Zn-ferrite (top), it consists of the doublet absorption similar to  $\text{Zn}_x\text{Fe}_{3-x}\text{O}_{4-\delta}$  ( $x = 0.993$ ) reported previously. The spectrum of the solid product after the solar decomposition reaction (bottom) comprises a broad sextet and a sharp doublet. It is almost the same as  $\text{Zn}_x\text{Fe}_{3-x}\text{O}_{4-\delta}$  ( $x = 0.751$ ). The sextet absorption was grown up along with reducing of the doublet absorption, since a part of  $\text{Fe}^{3+}$  ions in the Zn-ferrite were changed to  $\text{Fe}^{2+}$ . This indicates that if more than 40% of the Zn ion in Zn-ferrite is decomposed into metal Zn, the spectrum will give the usual two sextets exactly like magnetite.

#### 4. Conclusions

A unique low cost solar reactor dubbed the “sandwich reactor” proved successful concept for initial investigation of feasibility for carrying out the thermal decomposition of Zn-ferrite with concentrated solar radiation. It took less than 60 s for the Zn-Ferrite to reach an expected  $\text{O}_2$  generation reaction temperature of 1750 K in the insolation level of 840–980  $\text{W}/\text{m}^2$ . The solar decomposition of the Zn-ferrite commenced above 1500 K and its decomposition ratio at 1750 K was evaluated to be 40% from the lattice constant measurement and chemical analysis. The solar decomposition of Zn-ferrite can be readily promoted with increasing temperature. In effect constructing a “double glazed” window, adding a second smaller piece of quartz inside the main window of the sandwich reactor, the smaller piece of quartz in direct contact with the Ferrite powder experienced the highest temperatures, it was not forced out of shape. These results encourage further investigation for solar operation to enhance the solar decomposition ratio of the Zn-ferrite. The thermochemical cycles for  $\text{H}_2$  production with Zn-ferrite/Zn,  $\text{Fe}_3\text{O}_4$  system will be realized by the enhancement of solar decomposition of the Zn-ferrite.

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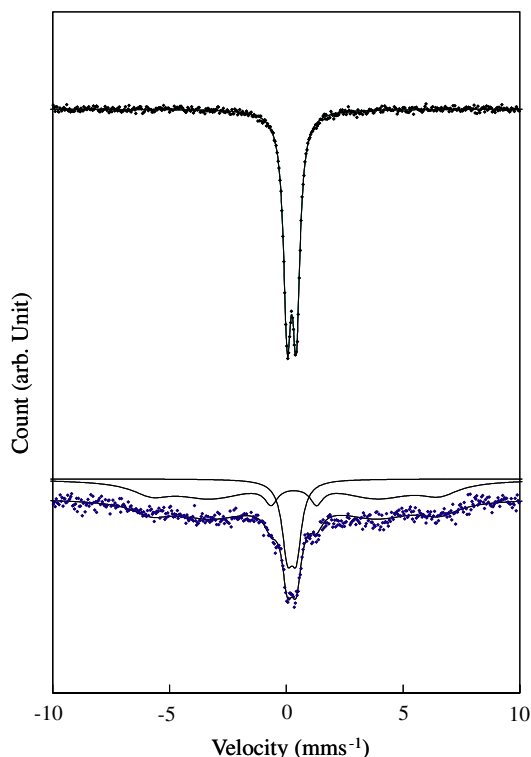


Fig. 8. Mössbauer spectrum of (a) Zn-ferrite, (b) Zn-ferrite after decomposition reaction, (---) least-square fit.

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