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Magnetic properties of Hagi ware

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Hagi ware originally consists of a mixture of two raw materials: Daido clay and Mishima clay. During its firing process, we observed a change in the magnetic properties of the iron oxide, Fe₂O₃. The magnetic moment of the Daido clay (which only contains a small amount of the Fe₂O₃ γ-phase) attains a maximum at a firing temperature of approximately 600°C, where a minor amount of the poorly crystallized Fe₂O₃ temporarily changes to the ferromagnetic γ-phase. Furthermore, the magnetic moment of the Mishima clay (which contains a large amount of the Fe₂O₃ γ-phase) decreases as the firing temperature increases, whereas the coercive field rapidly increases at firing temperatures above 1000°C. The magnetization curve of the Mishima clay that was fired at temperatures above 1200°C is characteristic of a two-component system consisting of a minor γ-phase and a major α-phase. The above-mentioned phenomena were also confirmed by XRD analyses. A series of experiments indicated that the firing of Hagi ware can be characterized as a transformation from the γ-phase of Fe₂O₃ to the α-phase of Fe₂O₃. This transformation is considered to contribute to the change from soft magnetism to hard magnetism of Hagi ware.

Key-words : Clay, Hagi ware, Iron oxides, Soft magnet, Hard magnet

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1. Introduction

In addition to Fe₃O₄, the iron oxide Fe₂O₃ is one of the most common magnetic materials, and it is generally classified into four phases, namely, the α, β, γ, and ε-phases. The α-phase and the γ-phase are abundant in the natural world, while the β- and ε-phases are rare and are therefore artificially synthesized. The α-phase (termed hematite) has a rhombohedral crystal structure, and it exhibits weak ferromagnetism at temperatures between −13 and 672°C as a result of the Dzialoshinskii-Moriya interaction. The coercive field (Hc) can have a value as great as 1 kOe, and the color of the hematite powder is red. The β-phase exhibits paramagnetism at room temperature and transforms into the α-phase at a temperature of 500°C. The cubic γ-phase (termed maghemite) exhibits ferrimagnetism. The basic crystal structure is the inverse spinel structure with some vacancy sites, and it theoretically has a saturation moment (Ms) per unit cell of 20μB (experimentally 17.36μB). Its magnetic transition temperature, Tc, is between 1093 and 1259°C, because the γ-phase transforms into the α-phase at temperatures above 400°C. Its coercive field, Hc, is one-tenth of that of the α-phase, and the γ-phase is representative of a soft magnet. The color of the maghemite powder is brown. Finally, the ε-phase does not naturally exist; however, its chemical synthesis, detailed structure and magnetic properties have been investigated recently. Thus, Fe₂O₃ is not only the subject of fundamental research but also has many possibilities for industrial applications. One of the applications of native Fe₂O₃ includes its use in the production of reddish-brown earthen-ware termed “Hagi ware”.

Hagi ware is one of the most famous ceramics in Japan, and it has an exceptional high amount of Fe₂O₃. We therefore considered that from a scientific viewpoint, Hagi ware is a good candidate to study natural iron oxides. Hagi ware consists of a mixture of light gray Daido clay [Fig. 1(a)] and bay Mishima clay [Fig. 1(b)]. The ratio of Daido clay to Mishima clay is usually 5:1 in the mass ratio. Both clays contain SiO₂, Na₂O, Al₂O₃, and Fe₂O₃, in addition to volatile constituents (mainly water), as seen in Fig. 2. The ratio of the volatile constituents within Hagi ware (the mixture of the Daido and Mishima clays) depends on the firing temperature. The percentage of volatile constituents within the Hagi ware is 11.6% in the dried state, 1.39% in the biscuit-fired state, and 0.00% in the glost-fired state.

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The magnetic properties of Daido clay can be attributed primarily to the poorly crystallized Fe$_2$O$_3$ and slightly to the γ-phase. However, the magnetic properties of Mishima clay can be almost entirely attributed to the γ-phase. Hagi ware is produced by performing biscuit-firing at approximately 700°C, followed by glost-firing on a mixture of Daido and Mishima clays at temperatures above 1200°C for more than one day. Following the biscuit-firing, the pottery is glazed and this determines the color of the final pottery. However, we are interested in the change in the intrinsic color of the mixed clays without glazing. Interestingly, the color of the sample becomes darker during the firing process between the temperatures of 35 and 700°C. Subsequently, by increasing the firing temperature to above 1200°C, the color prominently changes to white. As observed in Fig. 1(c), the sample fired at 700°C had the darkest brown color among all the samples that were fired (up to 1270°C). This was accompanied with a significant reduction in volatile constituents. It is well known that the origin of the brown color of Hagi ware (with glazing) can be attributed to Fe$_2$O$_3$. Given this background, it is interesting to note form a scientific perspective the correlation between the color of Hagi ware and its magnetic properties. However, the change in the magnetic properties of the Fe$_2$O$_3$ within the Hagi ware during the firing process has not been investigated at all. Both raw clays include a significant amount of Fe$_2$O$_3$, as shown in Fig. 2. Thus, we investigated the magnetic properties of Daido clay, Mishima clay, and their mixture (termed the Hagi ware), and confirmed via XRD analyses that the Fe$_2$O$_3$ phase changes within the two clays. Furthermore, we discuss the change in color that occurs during the Fe$_2$O$_3$ phase change.

2. Experimental procedures

We prepared the Daido and Mishima clays that are used in the pottery factories of Hagi ware. X-ray fluorescence analysis was conducted to characterize the chemical components of both clays; the results are presented in Fig. 2. Prior to firing, both clays were desiccated in a thermostat chamber at a temperature of 35°C for one week. Subsequently, both clays were fired at several temperatures using an electric furnace (referring to the actual firing conditions of the Hagi ware). The firing process depends on the maximum temperature, $T_{\text{max}}$. The time required to increase the temperature up to $T_{\text{max}}$ depends on $T_{\text{max}}$. The time required to fire up to $T_{\text{max}}$ of 700°C is 8 h, 8.75 h for 900°C, and 10 h for 1240°C. A series of firing procedures requires a period of approximately two days in total. Hereafter, $T_{\text{max}}$ will be described as the firing temperature, $T_f$.

We observed changes in the colors of both clays during the firing process: The Daido clay originally had a light-gray color, whereas during the firing process (up to $T_f = 600°C$) it tended to include a brown color. The clay had the darkest brown color at $T_f = 600°C$, subsequently returning to its initial color. However, the color of the Mishima clay during the firing process (up to $T_f = 1270°C$), which was originally reddish-brown, hardly changed. These results suggest that the color change of Hagi ware [Fig. 1(c)] is mainly due to the change in the color of the Daido clay.

We conducted magnetic measurements of Daido and Mishima clays fired at several temperatures using a commercial superconducting quantum interference device (SQUID) magnetometer. The magnetization ($M$) was measured at room temperature (set as 27°C) as a function of the magnetic field ($H$) in the range of 20 to $-20$ kOe. The total mass of the measurement sample was approximately 150 mg. The value of $M$ for the dried and fired clays was evaluated as the value per unit mass for each clay. The evaluation
of \( M \) per unit mass for \( \text{Fe}_2\text{O}_3 \) is only meaningful for the dried and
glost-
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ture Science, High Energy Accelerator Research Organization

the Photon Factory (PF) at the Institute of Materials Struc-
ture, High Energy Accelerator Research Organization

(KEK).\(^{15} \) The wavelength of the incident X-rays was 0.68841 Å.

3. Experimental results

3.1 Magnetization curves

Figures 3(a) and 3(b) show the magnetization curves of the
dried Daido and Mishima clays, respectively, at room temperature.
Both magnetization-curves suggest that there are both
ferromagnetic and paramagnetic components. The paramagnetic
component exhibits a linear relationship between \( M \) and \( H \); this
is owed to the poorly crystallized \( \text{Fe}_2\text{O}_3 \). By subtracting this
paramagnetic component (\( \chi H \)) (as shown by green lines in
Fig. 3) from the total \( M \), we can obtain the former ferromagnetic
contribution. Subsequently, when we compare these ferromag-
netic properties for all the compounds, a similar analysis will
be conducted.

The saturation magnetization (\( M_s \)) of the dried Daido clay was
1.6 \( \times 10^{-3} \) emu/g, corresponding to 8.9 \( \times 10^{-2} \) emu/g (\( \text{Fe}_2\text{O}_3 \)). Moreover, the \( M_s \) value of the dried Mishima clay was 3.7 \( \times 10^{-1} \) emu/g, corresponding to 3.1 emu/g (\( \text{Fe}_2\text{O}_3 \)). In the Mishima
clay, the linear contribution is small, and the magnetic properties
be mostly ascribed to the crystalline \( \text{Fe}_2\text{O}_3 \). When con-

considering the crystallinity of the \( \text{Fe}_2\text{O}_3 \), the Mishima clay is much
superior to the Daido-clay. A calculation based on the above facts
suggests that 98% of the ferromagnetic components of the dried
Hagi ware are owed to the Mishima clay. The coercive field (\( H_c \))
of the ferromagnetic components in the dried Mishima clay is
on the order of 0.1 kOe, and it is determined to consist of the
\( \gamma \)-phase of \( \text{Fe}_2\text{O}_3 \) (abbreviated as \( \gamma-\text{Fe}_2\text{O}_3 \)). One unit cell of
\( \gamma-\text{Fe}_2\text{O}_3 \) consists of eight \( \text{Fe}_2\text{O}_3 \) molecules, and the ideal
\( M_s \) of \( \gamma-\text{Fe}_2\text{O}_3 \) is 75.9 emu/g. Thus, only 4.1% of the \( \text{Fe}_2\text{O}_3 \) in the
Mishima clay consists of the good-crystalline \( \gamma \)-phase. As for the
Daido clay, the percentage of the good crystalline \( \gamma \)-phase of
\( \text{Fe}_2\text{O}_3 \) is approximately 0.12%.

Figure 4 shows the magnetization curves for the ferromagnetic
components of the Daido clays that were prepared at each \( T_f \). As
shown in Fig. 4(a), \( M_s \) hardly changes during the firing process
up to \( T_f = 300°C \). However, during the firing up to \( T_f = 700°C \),
\( M_s \) changes remarkably, and it achieves its maximum value at
approximately \( T_f = 600°C \). The hysteresis loop of the magnet-
ization curve for \( T_f = 500−700°C \) is still small, and \( H_c \) is on the
order 0.1 kOe. The above phenomena suggest that the \( \gamma \)-phase is
synthesized from the Daido clay during the biscuit firing process.

Thus, the Daido clay retains the characteristics of a soft magnet
even during the firing process.

Figure 5 shows the magnetization curves for the ferromagnetic
components in the Mishima clay prepared at each \( T_f \). Contrary to
the results of the Daido clay, the \( M_s \) value of the Mishima clay
systematically decreases as \( T_f \) increases up to 1000°C, remaining

![Fig. 3. Magnetization curves of the dried Daido (a) and dried Mishima clays (b) at room temperature. The observed magnetization (blue triangle), paramagnetic component (green line), and ferromagnetic component (red closed-circle) are displayed.](image)

![Fig. 4. Magnetization curves of the ferromagnetic components of the Daido clay prepared at each firing temperature (\( T_f \)).](image)
fairly constant at temperatures higher than 1000°C. In particular, the reduction of $M_s$ during $T_f = 700$–900°C is remarkable. The values of $M_s$ for the biscuit firing ($T_f = 700°C$) and the glost firing ($T_f = 1200°C$) correspond to 40 and 12%, respectively, of that for $T_f = 35°C$. The Mishima clay fired at $T_f = 1240°C$ has an $M_s$ value of $4.3 \times 10^{-2}$ emu/g, corresponding to $3.0 \times 10^{-1}$ emu/g (Fe$_2$O$_3$). Moreover, the Daido clay fired at $T_f = 1240°C$ has an $M_s$ value of $4.7 \times 10^{-3}$ emu/g, corresponding to $2.4 \times 10^{-1}$ emu/g (Fe$_2$O$_3$). The ferromagnetic components per unit mass of Fe$_2$O$_3$ for both clays fired at $T_f = 1240°C$ show similar values. Furthermore, the $H_c$ value of the Mishima clay hardly changes during the firing process up to $T_f = 1000°C$. However, it exhibits a considerable increase for $T_f > 1000°C$, and it exhibits an almost constant value for $T_f = 1100$–1270°C.

As mentioned above, the majority of the Fe$_2$O$_3$ in the dried Mishima clay is considered to consist of the $\gamma$-phase because of its ferromagnetic behavior (with $H_c \sim 0.1$ kOe). The decrease in the $M_s$ value suggests that as $T_f$ increases, the $\gamma$-phase changes to another phase. For $T_f > 1000°C$, the $H_c$ value exhibits an increase up to a few kOe. The results indicate that the majority of the $\gamma$-phase of the Mishima clay tends to transform into the $\alpha$-phase during the glost firing. In fact, the hysteresis loop for $T_f = 900$–1270°C is characteristic of a two-component system consisting of soft and hard magnets, and in the present case there is a mixture of the soft $\gamma$-Fe$_2$O$_3$ and the hard $\alpha$-Fe$_2$O$_3$.

**Figures 6(a) and 6(b)** exhibit the magnetization curves of the Hagi ware prepared at each firing temperature and those of the ferromagnetic components, respectively. As the $T_f$ value increases, the paramagnetic contribution increases, whereas the ferromagnetic contribution decreases. The ferromagnetic components for $T_f = 1220°C$ is characteristic of a two-component system consisting of soft and hard magnets, whereas those for $T_f = 1240$ and 1270°C are characterized as a single phase. The $H_c$ value for $T_f = 1240$ and 1270°C is over 5 kOe, which is attributed to the $\alpha$-Fe$_2$O$_3$ phase with good crystallinity.

**Figures 7(a)–7(c)** show the firing temperature ($T_f$) dependence of the saturation moment ($M_s$) and the coercive field ($H_c$) and the gradient of the line ($\chi H$) in the magnetization curves for the Daido clay, Mishima clay, and Hagi ware, respectively. First, for the $M_s$ value, it is interesting to note that the characteristic temperature for the maximum $M_s$ value for the Daido clay is close to the characteristic temperature for the decrease in the $M_s$ value for the Mishima clay and the Hagi ware. Secondly, as observed in **Fig. 7(b)** (for $H_c$), the prominent change in the Hagi ware is due to the change in the Mishima clay. The prominent magnetic phase of the Hagi ware following the glost firing consists of the $\alpha$-Fe$_2$O$_3$ phase of the Mishima clay. Finally, the paramagnetic component, owing to the poorly crystallized Fe$_2$O$_3$, tends to increase as the $T_f$ increases. Over the whole firing temperature range, the Mishima clay has a greater amount of paramagnetic components than the Daido clay. Looking at the data at approximately 600°C in **Fig. 7(c)**, the $\chi$ of the Daido clay slightly decreases, and an amount of the poorly crystallized Fe$_2$O$_3$ surely transforms into the $\gamma$-phase, suggesting a correlation with the increase in the $M_s$ value for the Daido clay, as shown in **Fig. 7(a)**. We cannot determine whether or not the $\gamma$-phase formed in the Daido clay transforms into the $\alpha$-phase during glost firing because of the small magnetic moment of the $\alpha$-phase.

**Figure 8** shows the $T_f$ dependence of the ferromagnetic moment of the Hagi ware, in addition to the ratios of the Daido and Mishima clays. The ferromagnetic moment of the Hagi ware exhibits a small jump at approximately $T_f = 500°C$ as a result of the increase in the ferromagnetic moment of the Daido clay. This characteristic temperature is consistent with the temperature at
which the color of the Hagi ware exhibits the darkest brown color. Indeed, the original material of Hagi ware is a mixture of Daido and Mishima clays (with a ratio of 5:1). According to a calculation, the magnetic moment of the Mishima clay at $T_f = 35,700,$ and $1240\,\text{°C}$ corresponds to approximately 98, 83, and 65%, respectively, of the $M_s$ value for the ferromagnetic component of the Hagi ware.

\[ M_s(35\,\text{°C}) = 98\% \quad M_s(700\,\text{°C}) = 83\% \quad M_s(1240\,\text{°C}) = 65\% \]

Fig. 7. $T_f$ dependence of the $M_s$, $H_c$, and $\chi$ values for the Daido clay, Mishima clay, and Hagi ware. The broken lines and curves are for visual guides.

Fig. 8. $T_f$ dependence of the observed and calculated ferromagnetic moment of the Hagi ware in addition to each ratio of the Daido and Mishima clays (in the case of the mixing ratio of the Daido clay and the Mishima clay = 5:1). The broken lines and curves are for visual guides.

### 3.2 XRD analyses

Figures 9(a), 9(b) and 10 show the XRD patterns for the Daido and Mishima clays, respectively. There are many diffraction peaks as a result of the inclusion of SiO$_2$, the oxides of Al and Si, the oxides of Al and Fe, the $\alpha$-phase of Fe$_2$O$_3$, and the $\gamma$-phase of Fe$_2$O$_3$. First, for the Daido clay, the components (except for Fe$_2$O$_3$) exhibit many diffraction peaks as seen in Fig. 9(a), the majority of which are for SiO$_2$. As shown in Fig. 2(a), the quantity of Fe$_2$O$_3$ included in the Daido clay is very little, so that it is difficult to recognize the diffraction peaks due to Fe$_2$O$_3$ in Fig. 9(a). With careful observation of the XRD patterns, we can distinguish between the $\gamma$-Fe$_2$O$_3$ peaks and the $\alpha$-Fe$_2$O$_3$ peaks. As seen in Fig. 9(b), the peaks labeled as the (220) plane index of $\gamma$-Fe$_2$O$_3$ at approximately $2\theta = 13.5\,\text{°}$ become more prominent at $T_f = 500$ and $600\,\text{°C}$ suggesting an increase in the amount of $\gamma$-Fe$_2$O$_3$. However, at $T_f = 700\,\text{°C}$, it is difficult to recognize the corresponding peaks. This behavior is consistent with the temporary increase in $\gamma$-Fe$_2$O$_3$ at approximately $T_f = 600\,\text{°C}$, as observed in the magnetic measurements.

Next, for the Mishima clay, many diffraction peaks due to $\alpha$-Fe$_2$O$_3$ are observed along with some small peaks that are due to $\gamma$-Fe$_2$O$_3$ in the dried state (see Fig. 10). The Mishima clay has a larger amount of Fe$_2$O$_3$ than the Daido clay, suggesting a correlation with the results shown in Fig. 2. In the dried material, the characteristic peaks of $\gamma$-Fe$_2$O$_3$ with the plane indices (220),
neither the volume ratio in the Mishima clay. The procedure for estimating the volume peak intensity with those of nine plane indices for analysis, (220), (422), (533), etc.]. By comparing the correspond-
ents for the dried clays mainly detected the magnetic properties whereas any evidence of the existence of the Fe2O3 can be observed. The Daido clay originally contains no Fe2O3 mixture per 1 kg of clay. Herein, we propose a tentative approach to manufacturing a soft magnet to hard magnet.

Mishima clay (with α-Fe2O3:γ-Fe2O3 = 10:1) is a necessary raw material in the production of Hagi ware and yields the brown color. The Daido clay originally contains no α-Fe2O3 even with firing. Herein, we propose a tentative approach to manufacturing Hagi ware without using Mishima clay. An artificially synthesized Fe2O3 mixture of α-Fe2O3:γ-Fe2O3 = 10:1 (at maximum, 20 g of the artificially synthesized Fe2O3 mixture per 1 kg of Daido clay) could be included in the Daido clay instead of mixing in the Mishima clay. Indeed, in order to evaluate the feasibility of this process, investigations with the use of glazes are required.

5. Conclusion

We investigated the changes in the magnetic properties of Hagi ware. The observed changes can be intrinsically explained by changes in the Fe2O3 phase. During firing at approximately 500–600°C, the Daido clay (which accounts for five-sixths of Hagi ware) exhibits a temporary increase in the amount of γ-Fe2O3 phase with good crystallinity. However, at temperatures above
1200°C, the ferromagnetic behavior is negligible. Furthermore, the magnetic moment of the Mishima clay (which accounts for one-sixth of Hagi ware) begins to rapidly decrease at approximately 500°C, which is close to the temperature of biscuit firing. Most of the γ-phase transforms into the α-phase at temperatures above 1200°C, which corresponds to the temperature of glost firing. This is exhibited as a type of transformation from a soft magnet to a hard magnet. This behavior is related to the change in the Fe₂O₃ phase, which was confirmed in the XRD experiments.

The remarkable change in the color of the Hagi ware (without glaze) at approximately 700°C is related to the increase in the amount of the γ-phase of Fe₂O₃ in the Daido clay, as well as to the rapid reduction of the volatile constituents.

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