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PRIMARY RESEARCH

Anti-copy and authenticity verification method for ceramic products: Development of new glass phosphor with two optical features

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Abstract

Multi-modal artifact metrics is an anti-counterfeiting technique and was created based on the concept of multi-modal biometrics. This method adds additives (functional materials) to the artifact during the manufacturing process to provide multiple feature information. Hence, the certainty of authenticity and difficulty of counterfeiting could be enhanced. We developed a new type of glass phosphor to provide two kinds of characteristic optical information (emission intensity and hue) into the artifact. This is a novel approach since up-conversion phosphors with a different hue and emission intensity by optical excitation at each observation point have never been reported. In this paper, we report the development of this glass phosphor, especially the production method and results of the fundamental experiments. The developed glass phosphor emitted as intended and demonstrated the validity of our approach.

I. INTRODUCTION

A. Background and Target

Ceramic products, such as high-class accessories (watches and bags), are made by renowned and well-known potteries or brand holders, are highly popular, and are likely to be sold at a high price. Forgers have been manufacturing imitations of these products and selling them through shopping and auction sites on the Internet in order to extend their market share [1, 2, 3, 4].

The manufacturing and distributing of counterfeit products does not only violate the intellectual property rights of potteries and brand holders and cost them a share of the income that they could have otherwise obtained, but also causes financial and psychological damage to the consumers who purchase them unknowingly. Hence, the existence of imitation ceramic products mimicking high-class accessories cannot be overlooked.

We developed a technique in order to verify the authenticity of ceramic products mechanically and to enhance the difficulty of counterfeiting genuine products, based on a concept called artifact metrics [5, 6, 7, 8]. In our previous study [9], we focused on a transparent glass phosphor which exhibited a one-peak emission wavelength in the near infrared wavelength band by optical excitation [10, 11, 12, 13]. Then, we proposed the idea of adding glass phosphor powder into the glaze or paint. This proposal has the following features: the powder does not affect the ceramic colors/patterns of these products, and is not toxic. The optical characteristic information can be extracted from ceramic products by using an infrared light source and contactless camera, which makes the counterfeiting of this information difficult.

The authors have also proposed a technology (multimodal artifact metrics) in order to enhance the accuracy of the artifact's authenticity and the difficulty of counterfeiting by

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adding multiple characteristic information to the artifact. The features of this technology were obtained by multimodal biometrics technology. In our previous study [14], we proposed a method of adding electric characteristic information (sheet resistance) and optical feature information (visible light emission from phosphor) into synthetic resin cards.

Since we aim to apply multimodal artifact metrics to ceramic products, in this study, we developed a new glass phosphor as the first step towards that direction. By utilizing this phosphor, the accuracy of the authenticity and the difficulty of counterfeiting could be enhanced, since each ceramic product has two optical characteristics, such as hue and emission intensity.

This paper is structured as follows: In Section 2, our proposal (development of new glass phosphor and application of multimodal artifact metrics) is outlined. In Section 3, the fundamental experiments (glass phosphor production) and results are presented. Considerations based on experimental results and conclusions are described in Sections 4 and 5, respectively.

B. Overview of the Artifact Metrics

In artifact metrics technology, each artifact's authenticity is verified mechanically by utilizing unique information (characteristic information) extracted from each artifact [15, 16, 17, 18]. Hence, the concept of this technology is the same as the biometrics. The difficulty of counterfeiting is established on the basis of technological evidence, by which the characteristic information held by the genuine product seems to be difficult to copy. Characteristic information is formed spontaneously and randomly during the manufacturing process and cannot be formed intentionally even by the manufacturers of genuine products. Hence, the difficulty of counterfeiting cannot be decreased if the formation method of characteristic information is accessible to the public.

Microscopically, each artifact has different characteristic information, such as tiny roughness and color shade, as observed by a microscope. However, specifying the area photo by the registration phase and the area photo by the verification phase seem to be time-consuming, since the area observed by the microscope is quite small [9].

Hence, in artifact metrics, a method to form unique and easy-to-extract characteristic information in the artifact was utilized. Specifically, additives with physical features were added during the manufacturing process. The particles of the additives are distributed randomly and non-uniformly and fixed in the artifact. Their degree of distribution reflects the characteristic information. Sensing devices, which can extract the additives' physical features, are used when extracting characteristic information. Table 1 shows the physical characteristics of material added in the manufacturing process and the characteristic information extracted from them.

Figure 1 shows an overview of the system (artifact metric system) that uses the artifact metrics. Indicated two phases are almost the same as those of biometrics. In this system, characteristic information is extracted from each artifact before it is shipped, and the information is stored in a secure database. To verify the authenticity of an artifact, the system extracts the characteristic information from it and compares this information with the registered feature information from the secure database.

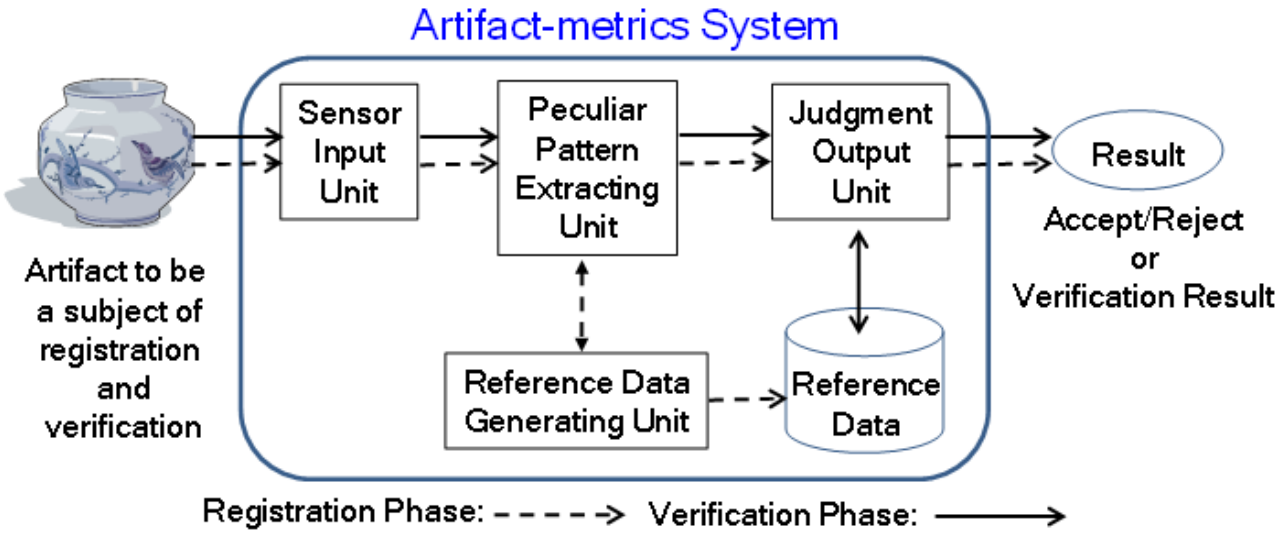

Fig. 1. Overview of artifact metric system

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TABLE 1
PHYSICAL CHARACTERISTICS AND EXTRACTED INFORMATION

Physical Characteristics	Extracted Feature Information
Optical characteristics	Particles' optical characteristics (reflection, transmission, infraction, and fluorescence) and their degree of distribution reflect the characteristic information, which is extracted by sensors that can detect light intensity.
Magnetic characteristics	Particles' magnetic characteristics (attraction and repulsive force) and their degree of distribution reflect the characteristic information, which is extracted by sensors that can detect a change in magnetism.
Electrical characteristics	Particles' electric characteristics (electrical charge) and the degree of distribution reflect the characteristic information, which is extracted by sensors that can detect the quantity of electric charge.
Vibration characteristics	Particles' vibration characteristics (sonic waves) and the degree of distribution reflect the characteristic information, which is extracted by sensors that can detect sonic waves.

C. *Overview of Multi-modal Artifact Metrics*

Characteristic information extracted from artifacts can be changed depending on the environmental circumstances during extraction (such as temperature, humidity, and position of the artifacts relative to the sensing devices). However, even in such situations, the artifact metric system should be able to verify authenticity stably and correctly based on the strong correlation between the characteristic information registered in the database and the information features extracted during verification.

There is an approach to increase the number of characteristic information in order to find strong correlations between both registered and extracted information [9]. This approach can be categorized in two ways. The first method (approach 1) focuses on the additives of one physical feature and extracts two or more types of characteristic information from the artifact. For example, in our previous study [9], we focused on the glass phosphor's optical feature and proposed an idea of utilizing two distributions (emission spectrum and emission intensity distribution) as the characteristic information, since these distributions are reflected by the glass phosphor's particle size and degree of dispersion welded on the surface of ceramic products.

The second method (approach 2) adds two or more additives with different physical features to the artifact, from which it extracts two or more types of characteristic information. For example, in our previous study [14], we proposed the idea of forming thin film inside synthetic resin cards by using a conductive polymer paint with electrical features, and infrared up-conversion phosphor powder with optical features. This method utilizes the size and dispersion of phosphor particles in the thin film, which are reflected by emission intensity and sheet resistance. This information could be used as the characteristic information.

TABLE 2
COMPARISON OF TWO APPROACHES

	Approach 1	Approach 2
Advantages	Low probability of affecting the artifact's moldability and physical strength.	Increases the number of extractable feature information.
Drawbacks	Limited with regard to the number of extractable feature information.	High probability of affecting the artifact's moldability and physical strength.

The differences of both methods are presented in Table 2. Although the former method is limited with regard to the amount of characteristic information extracted from the artifact, it could reduce the quantity of the material for the total amount of artifacts. The latter method could increase the number of characteristic information more than the former one; however, it could also affect the artifact's moldability and physical strength, since the material quantity of the total amount of artifacts increases.

II. INVESTIGATION APPROACH

A. *Previous Method*

In our previous study [9], we focused on the glass phosphor that emits near infrared light with a peak wavelength

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of 1,000 nm, when it receives a specific peak wavelength (800 nm) of near infrared light. We proposed a method to add this powder into the glaze or paint. This phosphor is called the down-conversion phosphor, since the emission wavelength shifts to the opposite direction (or long wavelength side) of the optical excitation wavelength.

In the furnace, the glass phosphor particles melt with the substance of the glaze or paint, when the inner temperature exceeds 1,000° C, and they are subsequently vitrified and welded onto the surface of the ceramic products, when the inner temperature decreases below a specific temperature. Although the color of the glass phosphor is pale blue, it is also transparent; therefore, it is difficult to determine the existence of welded glass phosphor particles on the surface of ceramics, and does not affect the products' colors/patterns if the amount of glass phosphor is very small.

The size and degree of dispersion of the welded phosphor particles are determined randomly in the furnace, and these are reflected by the difference of infrared light emission intensity, when the excitation light is irradiated on the surface of the ceramic products. Figure 2 shows the images (infrared spectrum images) by which the infrared light emission is captured by optical excitation. Infrared light emission is expressed by whitish spots. The verification of the product's authenticity was done mechanically by using an image matching technique that utilizes images, such as Figure 2, as the characteristic information.

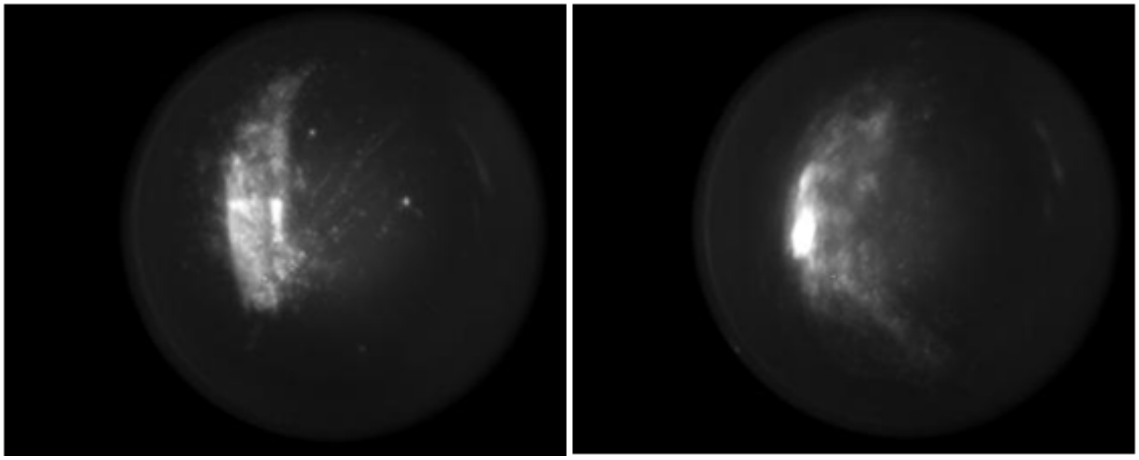

Fig. 2. Infrared spectral images

B. Challenges

As described in Section 3, in our previous study [9], the idea of utilizing two types of characteristic information (emission spectrum and emission intensity distributions) was proposed because it is reflected by the welded glass phosphor particle size and degree of dispersion on the surface of the ceramic products. Hence, since multimodal artifact metrics could be applied to ceramic products, this method could ensure the product's authenticity and difficulty of counterfeiting, more so than artifact metrics (described in the previous section), which only use one type of feature information. However, in this method, a complicated and expensive optical system, such as infrared hyper spectrum cameras, is required in order to extract the two distributions.

C. Solution

Visible light cameras can capture the light emission spectrum (wavelength and corresponding color), and the weak/strong visible light (light emission intensity) in the visible light band. This means that they can capture these two types of optical feature information simultaneously.

Visible light cameras are simple optical systems and available at affordable prices. As a way to apply multi-modal artifact metrics to ceramics, we propose using visible light cameras as optical systems. We also conceived the idea of developing a new glass phosphor, with a two-peak wavelength in the visible light band, by optical excitation (Figure 3 and Figure 4). Similar to the previous phosphor [9], this has the potential of leaving the ceramics' colors/pattern unaffected, if the amount of the phosphor particles welded onto the surface of the product is very small, since determining it seems to be difficult. It also has high affinity for glaze and paint, because glaze and paint share the same glassy feature.

D. Development of Novel Glass Phosphor

Since the novel glass phosphor has a two-peak wavelength in the visible light band (Figure 4), it shows different light emissions based on the locations where the excitation light

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is irradiated. As shown in Figure 3, for example, the red color could be observed at observation point A, while the green color could be observed at observation point B. If the yellow color can be observed, this means that the red and green lights are emitted at that point. The reason why the emission difference occurs based on the observation point is the difference of the ratio of the number of ions from the multiple rare earth oxides contained in the glass phosphor.

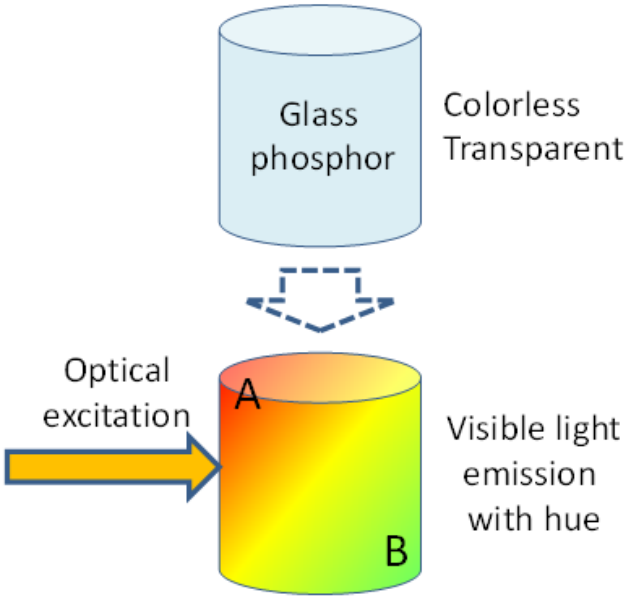

Fig. 3. New glass phosphor

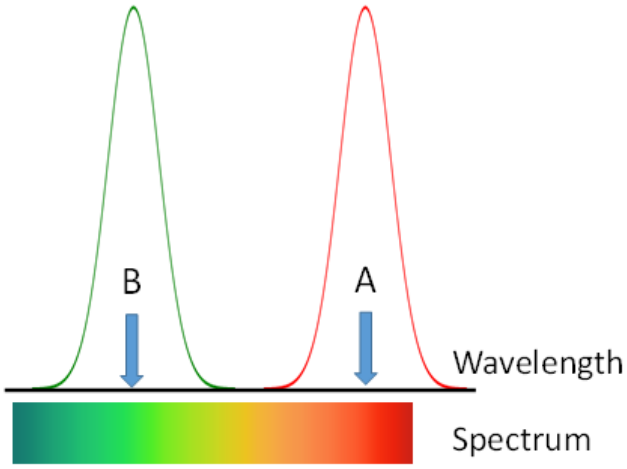

Fig. 4. New glass phosphor

Similar to our previous study [9], we chose an approach of mixing the glass phosphor powder with glaze and paint. Two types of feature information, namely, emission spectrum distributions (hue) and emission intensity distributions, could be extracted from each observation point, when the glass phosphor particles are welded onto the surface of the product, provided that the unevenness of the number of ions was retained.

Generally, the up-conversion phosphor (emission wavelength shifts to the short wavelength side rather than the optical excitation wavelength), which exhibits one-peak wavelength in the visible light band and requires infrared light as the light source, was developed with the aim of having one sharp peak wavelength (or single color). Thus far, our approach seems to be novel, since developing an up-conversion phosphor with a two-peak wavelength by changing the ratio of the rare earth oxide has never been reported.

An approach exists for developing the down-conversion glass phosphor with the same features utilizing an Ultravi-

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olet (UV) light source. However, UV light could affect the colors/pattern of the ceramic, while irradiating the surface of the ceramic products. Hence, in this study, we developed an up-conversion phosphor which utilized an infrared light source.

III. EXPERIMENTS

A. Searching Appropriate Composition (Rare Earth Oxide)

The glass phosphor's colors and transparency and the emission light colors with optical excitation were determined by the glass phosphor's types and ratio, as well as the base glass material's composition. The target glass phosphor contained two types of rare earth oxides (Yb_2O_3 and Er_2O_3), while the previous glass phosphor [9] consisted only of one rare Earth Oxide (Nd). These compositions could generate points in the glass phosphor where the number of ytterbium and erbium ions would be spontaneously uneven during the firing process, and this phenomenon could provide different emission spectra (hue) and emission intensity at each different observation point. The ratio of the number of ytterbium and erbium ions could change the emission spectra (hue), while the number of erbium ions could change emission intensity. In our experiment, we changed the composition of the base glass material (ZnO, Sb_2O_3, and GeO_2) and rare earth oxides little by little, weighted the reagents carefully, placed them in crucibles, and fired them in a furnace. Subsequently, we observed the luminescence of each sample by using optical excitation. We repeated these processes in order to estimate their appropriate compositions.

B. Searching Appropriate Composition: Base Glass Material

Our original plan was to use the base glass material composed mainly of ZnO (or 50ZnO-50H_3BO_3), while using Yb_2O_3 and Er_2O_3 as the rare earth oxides. Although the glass phosphor developed from these materials had high transparency and did not show any discoloring, the emission intensity was quite low due to compatibility with rare earth oxides. Hence, in this study, we decided to use 10ZnO-45Sb_2O_3-45GeO_2 as the base glass material, based on our previous study, since it was effective in improving the emission intensity.

Chemical formula of targeted glass phosphor is as follow;

$$XYb_2O_3 - YEr_2O_3 - \left(\frac{10ZnO-45Sb_2O_3-45GeO_2}{Base\ \ glass\ \ material}\right)$$

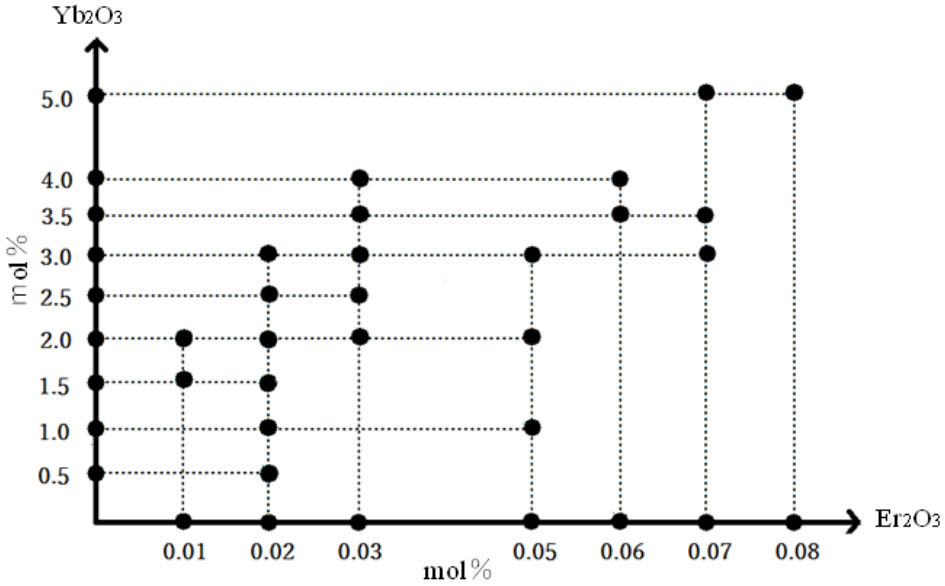

Fig. 5. Value of mol % and number of samples

C. Sample Making

Glass phosphors are made by mixing two types of rare earth oxides and the base glass material in the crucible and firing it for approximately 20 minutes at 1,230° C in the furnace. Since we used a small furnace while firing, we set the weight of each glass phosphor sample to 5 g. Figure 5 shows the chemical formula of the target sample. The procedures for making the glass phosphor are as follows;

(1) Define the value of mol % of each ytterbium oxide and erbium oxide as X and Y, respectively, and calculate the corresponding weight. X and Y correspond to the red-colored letter indicated in Figure 5.

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(2) Weigh the reagents based on the weight calculated in step (1). Next, fire the crucible containing the reagents in the furnace.
(3) Irradiate the excitation light to the sample made in step (2). When the difference of the hue and emission intensity is observed by naked eye at any location, set another X and Y close to the initial X and Y in order to confirm the existence of stronger light emission in the vicinity of X and Y by optical excitation.
(4) Return to step (1).
The reason for conducting the above procedures is to efficiently make glass phosphor with a two-peak wavelength and to easily determine the factors affecting the emission spectrum, emission intensity, and the phosphor coloring.
Figure 5 shows the values of X and Y and the number of samples. The vertical axis shows the mol% value of erbium oxide, while the horizontal axis shows the mol% value of the ytterbium oxide. In this experiment, we made 22 samples.

D. *Fluorescent Observation*

Samples with such visible light emission could be observed by naked eye, and were set in the optical system (Figure 6) in order to shoot visible light emission. We used a commercially available single-lens reflex camera (SONY α7s) with an IR cut filter. A sample with a distinct hue at each observation point and a stronger visible light emission, in comparison to the other 21 samples, is shown in Figure 7. This sample consists of 5.0 mol% of ytterbium oxide and 0.08 mol% of erbium oxide. Although this sample had two colored parts (black and white), we could observe the difference of the fluorescent color at any observation point. Figure 8 shows the fluorescence at the black part, while Figure 9 shows the emission at the white part.
We observed the yellow color at observation point (1) and (4), the red color at observation point (2), and the orange color at observation point (3), respectively (see Figure 8). We also observed the green color at observation point (A), the red color at observation point (B), the orange color at observation point (C), and the yellow color at point (D), respectively (Figure 9).

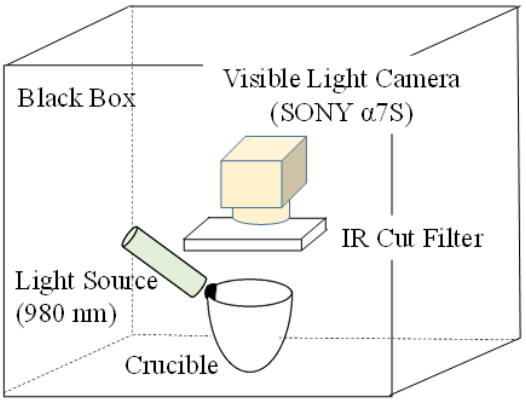

Fig. 6. Photo of visible light emission

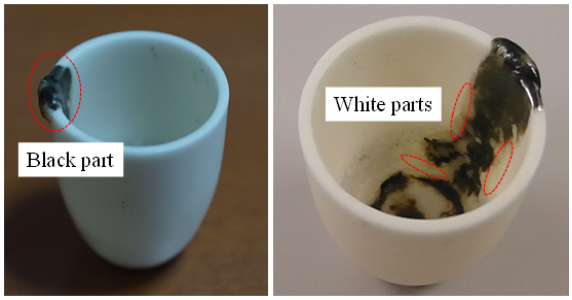

Fig. 7. Glass phosphor sample

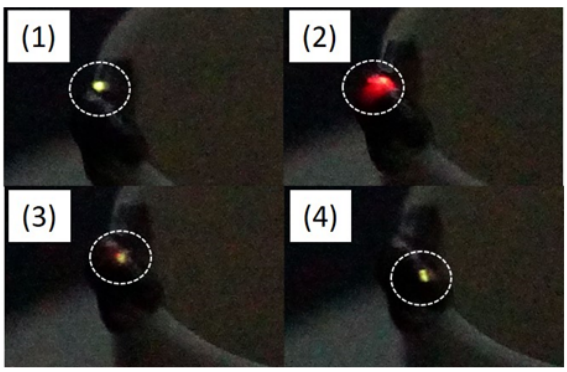

Fig. 8. Visible light emission (black part)

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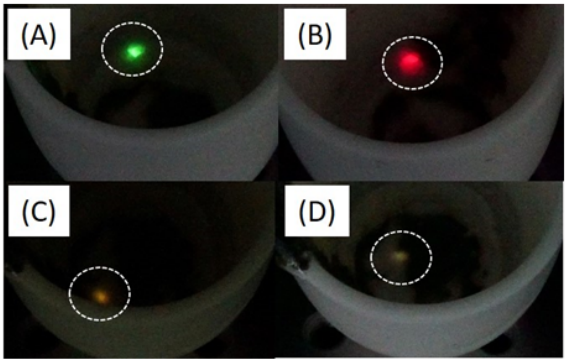

Fig. 9. Visible light emission (white part)

E. Observation of Emission Spectrum and Intensity

Figure 10 shows an optical system that can measure the emission spectrum and intensity. A spectroscope measures the emission spectrum and intensity of visible light and outputs the measurement results to PCs.

Figure 11 shows the measurement results at observation point (1) shown in Figure 8, while Figure 12 shows the measurement results at observation point (2) in Figure 9. The vertical axis shows the emission intensity, while the horizontal axis shows the wavelength. The red and green lights are shown in Figure 13. Naked eyes will observe the yellow light by mixing two light colors. Figure 13 shows the red-light emission whose intensity is stronger than that shown in Figure 11. These results correspond to the image shown in Figure 8.

The measurement results of observation point (A) are shown in Figure 9 and the measurement results of the point (B) are shown in Figure 15. The green light is shown in Figure 14, while the red light is indicated in Figure 15. Each emission intensity is stronger than other points.

From these results, we could say that a glass phosphor with different hue and emission intensity at any observation point was created.

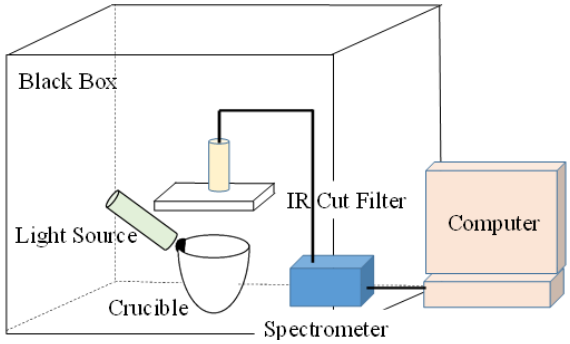

Fig. 10. Spectrum and emission intensity observation

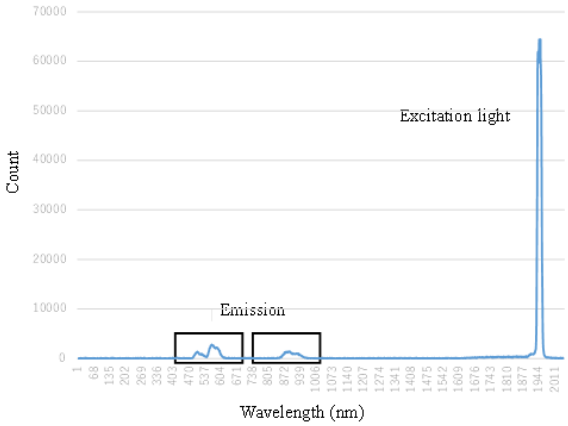

Fig. 11. Emission spectrum (observation point (1))

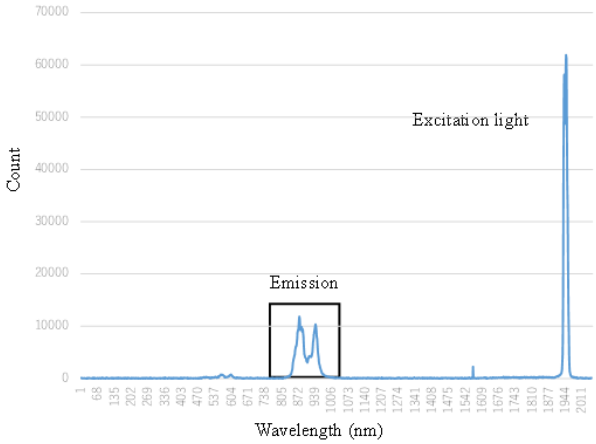

Fig. 12. Emission spectrum (observation point (2))

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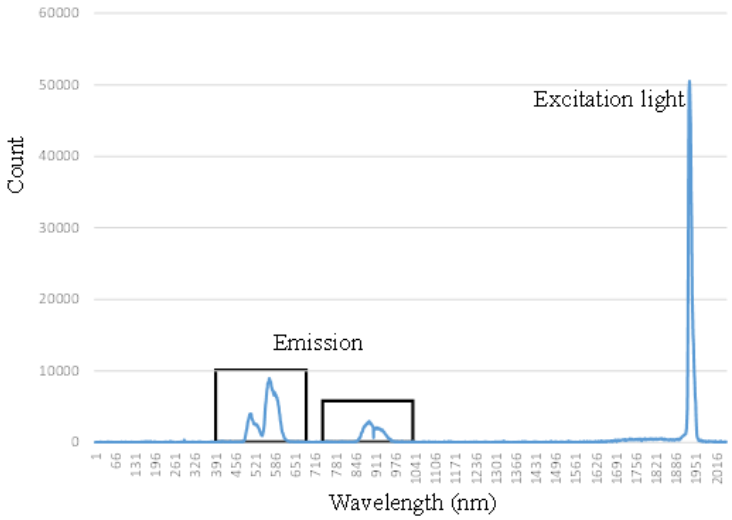

Fig. 13. Emission spectrum (observation point (A))

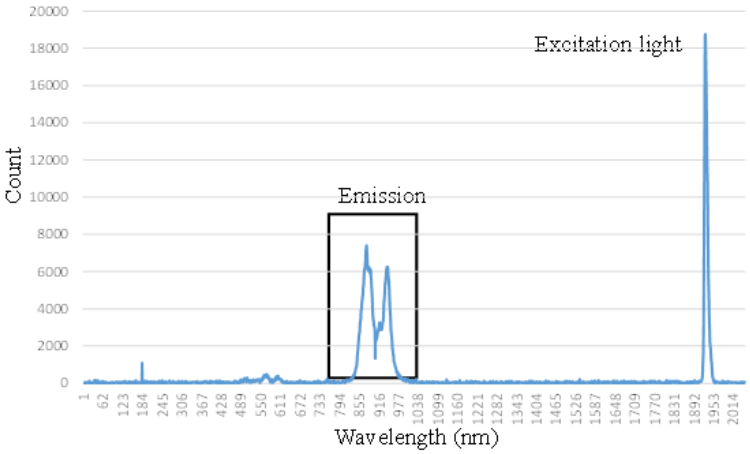

Fig. 14. Emission spectrum (observation point B)

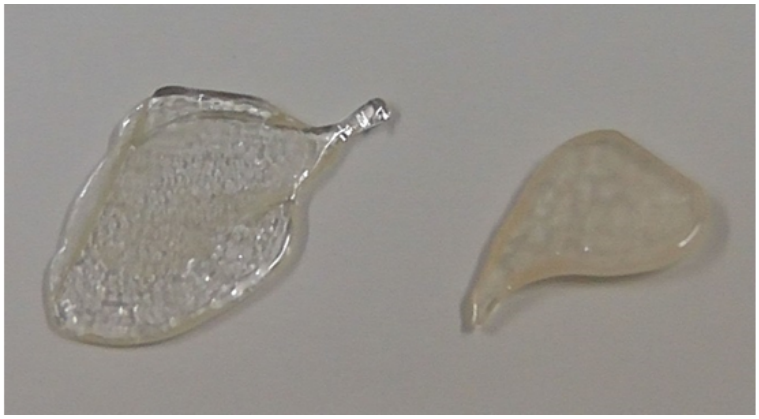

Fig. 15. Other samples differing

IV. CONSIDERATIONS

A. Coloring Removal

Although the glass phosphor introduced in Section 3 (consisting of 5.0 mol % of ytterbium oxide and 0.08 mol % of erbium oxide) has distinct hue and strong emission intensity in comparison to other samples, it also has black-colored parts. On the other hand, other samples (shown in Figure 15) do not have black-colored parts, but still, a distinct hue and strong emission intensity could not be observed. Two reasons are considered for the existence of black-

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colored parts; namely, the increase in the amount of rare earth oxides and the high firing temperature. The former is the proneness obtained from our experiments, while the latter is based on the phenomenon where the black color can be seen by the reduction occurrence, due to the high firing temperature.

Since our target glass phosphor was colorless and transparent, we should remove the color. Hence, we will create it by reconsidering parameters, such as the amount of rare earth oxide, firing time, and temperature.

B. Improvement of Emission Intensity

Although the glass phosphor introduced in Section 3 (consisting of 5.0 mol % of ytterbium oxide and 0.08 mol % of erbium oxide) had stronger emission intensity in comparison to other samples, it was weaker than the glass phosphor used in our previous study [9].

Two approaches were considered as a method of improving emission intensity. One was to remove the fine particles in the glass phosphor. Fine particles absorb the light emitted by glass phosphor, such that emission intensity can be brought up to natural levels by removing them.

The other approach was to reconsider the content of the glass phosphor; namely, rare earth oxides and base glass material. The emission intensity of the glass phosphor is determined by the chemistry between the rare earth oxide and the base glass material. In our experiment, we used $10ZnO\text{-}45Sb_2O_3\text{-}45GeO_2$ as the base glass material and ytterbium and erbium as the rare earth oxides. However, the intensity could be improved by using other potential materials.

V. CONCLUSION

A. Summary

We proposed a novel glass phosphor with a two-peak wavelength in the visible light band by optical excitation. This material could be used by ceramics, for the purpose of authenticating each product through the application of our proposed technique: multimodal artifact metrics. Two types of feature information (hue and emission intensity) could be extracted from different observation points by welding the glass phosphor powder onto the surface of the product during the manufacturing process, while retaining the unevenness of ion concentration. In our experiment, we created glass phosphor samples by changing the blending ratio of erbium and ytterbium oxides in order to estimate the appropriate composition. As a result, we created a glass phosphor with distinct hue and emission intensity.

B. Future Work

The glass phosphor created in our experiment had black color and its emission intensity was weaker in comparison to the glass phosphor created in our previous study [9]. In future work, we will reconsider parameters, such as composition, firing temperature, and time, in order to improve it.

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