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Characteristic Analysis of Sintered Glaze on Copper Metal Material Surface

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***Abstract*—Glaze's many properties, such as strong acid and alkali resistance, non-conduciveness, insolubility, and gas-impermeability, make it a great material to be used as protective membrane to prevent oxidation. The purpose of this study is to examine the Sintered Copper Sheet Metal Glaze Layer Technology. First, to find the best copper sheet metal glaze sintering method, Taguchi $L_{16}(4^5)$ orthogonal array was used to identify and determine sintering organically by examing different temperatures, firing times, the thickness of the copper metal. Then salt spray test was used to seek the strength of the glaze's mechanical properties. By applying the optimal design parameters, we identified the significant control factors that determine the optimal sintered ceramic layer of the surface structure. Findings in this study could be further used to seek the optimal composition of metal and glaze in order to improve the yield of glaze related products.**

***Index Terms*- Glaze, Copper Metal Material Surface, Taguchi method, Salt spray test**

I. Introduction

There are numerous wares in modern people's daily life benefiting from "glaze", such as bowls and plates for meals, noggin and floor tiles for architecture, ceramic vases and wall paintings for decoration, ceramic pots and enamel pots for kitchen, even sanitary equipment such as washbasins, bathtubs and closestool. There is a layer of glaze on the surface of these wares, which not only brings conveniences for people's daily life, but also makes people's life colorful.

With rapid development in flourishing science and technology, glaze has gained increasing attention in a wide range of applications. For instance, it has been applied to building, public art, interior design, industrial arts and product design. Many industrially mass-produced metal types of enamel have been developed, such as external wall of buildings, office furniture, external wall of rapid transit system, decorative wall of metro system, household electric appliances and so on. The glaze is a vitrified material; it is thin and mean silicate mixture without an exact melting point, but a melting range [1]. Metal enamel process is to draw colorful glaze on metal surface, at the end of which the metal surface is covered with a layer of glass glaze or multiple glazes by firing. The name of enamel has two fold meanings. The technique of decorating glaze on smithcraft products is called enamel and it is the generic terms of enamel process. The material of glaze implementation is also known as enamel. For instance, enamel may be used to decorate metal surface and replace expensive jewels. Ultimately, a characteristic ornament, enamel process is developed from this material, which is characterized by a variety of color and fastness [2]. The enamel wares are usually of smooth surface and high hardness and they are unlikely to be scratched or worn. Additionally, they have no strange odor and they are resistant to high temperature and corrosion, not to mention their long lifetime [3], [4]. The metal enamel process is often operated at the temperature of 750 ℃-850 ℃ [5], [6]. The basic glaze fired shows white or transparent brightness. Addition of metal oxides can produce diversified glaze colors. The fired enamel has similar characteristics to that of intermetallic and glass. They are also corrosion resistant [7]. Very few comprehensive studies on the technique of sintering glaze on metal can be found in the literature. It is still challenging to fully understand the firing effect. This study uses Taguchi method to examine the correlation among different variables, e.g., kiln temperature of glaze, firing time and copper metal thickness and the mechanical properties.

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Ⅱ. Research Objectives

The enamel process of sintering "glaze" on copper metal is very sensitive to external factors that could influence the quality of enamel discharged from the kiln. Each of the processes, e.g., grinding, cleaning, enamel powder, painting layer by layer, kiln temperature control and kiln feed time should be paid close attention to the final product, could otherswise fail at last.

This study uses Taguchi method to examine the correlation among different variables, e.g., kiln temperature of glaze, firing time and copper metal thickness and the mechanical properties. The salt spray is used to test adhesion strength after sintering and the effect of hardness is also tested to search out the optimal sintering method to provide related creators with steadier, more convenient and easily operated "glaze" sintering data, promoting the application of the existing glaze. In addition, the type and dosage of glaze shall be adjusted to design different products. For instance, for the commercially available glaze, its expansion coefficient is smaller than porcelain, and its melting point is lower than porcelain. By doing so, it could adhere to the ware surface completely and enhance the ware's esthetic appearance.

Ⅲ. EXPERIMENTAL DESIGN AND METHOD

A. Experiment setup

To control the process of sintering glaze on copper metal plate, the experimental design controls the glaze mesh, copper plate thickness and firing temperature. The glaze screening mesh numbers are chosen as 60, 90, 120 and 150, respectively (larger mesh number represents finer glaze particles). The metal sample of copper plate in dissimilar thickness (1.0, 1.5, 2.0, and 2.5 mm) is smeared with glaze, fired at four different temperatures (700 ℃, 750 ℃, 800 ℃, and 850 ℃). The heating curve rises by 200 ℃ per hour. Upon the preset sintering temperature is reached, the temperature is kept for 2 to 12 minutes before natural cooling.

B. Five stages of glaze sintering

(1) The glaze is dried, but not attached to metal in grains.

(2) The glaze begins to dissolve, partially granular, partially smooth.

(3) Most of glaze has dissolved, which is the cellulite stage.

(4)The glaze has dissolved completely; appropriate color is shown, but uneven.

(5) The glaze has reached sufficient firing time at appropriate temperature, with smooth surface.

C. Definition and method of salt spray test

The salt spray test uses manually simulated salt mist environment created by salt spray test equipment to check the corrosion-resisting properties of products or metal material. It can be categorized as two major types, one is natural environmental exposure test, and the other is artificially accelerated salt mist environment test. The manually simulated salt mist environment test makes a salt mist environment manually in the volume space of a test equipment, e.g., salt spray chamber, to check the salt spray resistance of test products. In comparison, the salinity of chloride in the salt spray environment can be several times or even tens of times of the salt spray content in general natural environment, accelerating the corrosion greatly. The result of salt spray test is obtained much earlier. If a production sample is tested in natural exposure environment, its corrosion may take up to one year. Similar results, however, can be obtained only with 24 hours in the manually simulated salt spray environment.

Ⅳ. Results and Discussion

A. Sintering adhesion strength

The Electronics Testing Center, Taiwan (ETC) is commissioned to carry out the salt spray test. The actual operation follows CNS8886 specifications and the test conditions are described as following: Temperature: 35 ℃, Brine solution concentration: 5.0 % (weight ratio), PH value (mean value): 6.5 (standard 6.5~7.2), Spray capacity (mean value): 1.7 ML/hr (standard 1.0 ML~2.0 ML/hr), Testing time: 48 hours, Measurement environment: temperature 23 ℃~24 ℃, Relative humidity: 54 %~56 %. The SEM electron beam is used and the detectability is about 1.8 A to observe fine structure and analyze the crystal structure and chemical composition. When the glaze is sintered on the copper plate and 16 experimental samples have passed the salt spray test, the experimental sample Code 1444 is preliminarily identified as the worst, and Code 3134 as the best, and the microstructure is magnified 10000 times through SEM (Code 1444 dotted line indicates obvious glaze peeling gap, Code 3134 dotted line indicates good glaze bonding), as shown in Figure 1.

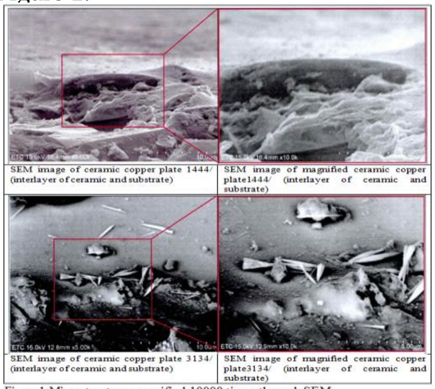

Figure1 Microstructure magnified 10000 times through SEM

B. Taguchi experiment and experimental data calculation

This experiment uses Large-the-Best characteristic; the salt spray test RN (ranking number) uses Large-the-Best characteristic. The S/N ratio is drawn into auxiliary table (Table 1 and Table 2) and response graph (Figure 2). According to Table 1 and Figure 2, the quality is best when the control factors are in the combination of A1, B1, C3, D1 and E2. The order of influence is C3, A1, D1, B1 and E2. The predictive value of RN can be calculated using 'half guidelines'. The general half guidelines use the first half of influencing factors as important factors, and the other factors can be regarded as unimportant factors B and E can be regarded as unimportant factors and the prediction equation is described below:

S/N prediction value = ý + (AA-ý) + (BB-ý) + (CC-ý) + (DD-ý)... (1)

where AA, BB, CC, and DD in Eq. (1) represent optimum combination, ý represents the mean value of S/N ratio. The three most important factors in this experiment are selected according to half guidelines, which are C3, A1, and D1. Therefore, the prediction value of the optimum design ratio, S/N, is calculated as follows:

18.85 + (19.73-18.85) + (19.50-18.85) + (19.46-18.85) = 20.99

The prediction value of S/N is close to the experimental value of Group 3. It is larger than the other 15 groups of experiment in Table 1, denoting that the quality characteristic can be improved.

Table 1 S/N ratio value

Experimental factor	Thickness (mm)	Mesh (mm/in)	Sintering temperature (℃)	Sintering time (min)	Room temperature (℃)	Salt spray RN	S/N ratio
No.	A	B	C	D	E		
1	1.0	60	700	12	25	9.3	19.37
2	1.0	90	750	8	30	9	19.08
3	1.0	120	800	4	35	10	20.00
4	1.0	150	850	2	40	9.5	19.55
5	1.5	60	750	4	25	8	18.06
6	1.5	90	700	2	30	7	16.90
7	1.5	120	850	12	35	9.3	19.82
8	1.5	150	800	8	40	9.5	19.55
9	2.0	60	800	2	25	9.8	19.82
10	2.0	90	850	4	30	9.5	19.55
11	2.0	120	700	8	35	8	18.06
12	2.0	150	750	12	40	9	19.08
13	2.5	60	850	8	25	9.3	19.37
14	2.5	90	800	12	30	9.5	19.55
15	2.5	120	750	2	35	7	16.90
16	2.5	150	700	4	40	7	16.90
Mean value						8.79	18.85

Table 2 S/N list

	Thickness (mm)	Mesh (Mesh/in)	Sintering Temperature (℃)	Sintering Time (min)	Room Temperature (℃)
Experiment No.	A	B	C	D	E
Level 1	19.50	19.16	17.81	19.46	18.85
Level 2	18.59	18.77	18.28	19.02	18.91
Level 3	19.13	18.70	19.73	18.63	18.84
Level 4	18.18	18.77	19.58	18.30	18.81
EFFECT	1.32	0.46	1.92	1.16	0.10
RANK	2	4	1	3	2

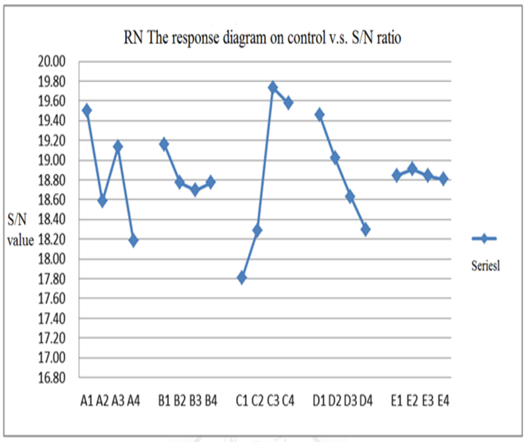

Figure 2 RN control factor versus S/N ratio response graph

3. Variance analysis of application software MINITAB

One-way ANOVA (analysis of variance) of MINITAB software is used to calculate p-value (or significance value, significance level ≤ 0.05) aiming at H_1, the sample value H_0 is true reliability. The most influencing three factors in the experiment are the above-mentioned C3, A1, and D1 according to half guidelines, substituted in the one-way ANOVA of MINITAB software as per RN. P-value of sintering temperature to RN is shown in Figure 3.

One-way ANOVA: RN versus sintering temperature (S. Temp)

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Source                 DF      SS     MS     F      P
Sintering temperature   3   9.692  3.231  5.93  0.010
Error                  12   6.538  0.545
Total                  15  16.229

S = 0.7381   R-Sq = 59.72%   R-Sq(adj) = 49.65%

                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level  N   Mean  StDev   +---------+---------+---------+---------
700    4  7.825  1.090   (-------*-------)
750    4  8.250  0.957       (-------*--------)
800    4  9.700  0.245                      (-------*-------)
850    4  9.400  0.115                   (-------*-------)
                         +---------+---------+---------+---------
                        7.0       8.0       9.0       10.0

Pooled StDev = 0.738
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Figure 3 One-way ANOVA: RN versus sintering temperature (S. Temp)

This study discusses the development of glaze sintering on copper metal plate products. There are 16 samples of copper metal made by experiment, sintered under different plate thicknesses, meshes, temperatures and times and magnified 10000 times by SEM to observe the microstructure and EDS. The copper plate and glaze elementary composition are analyzed and a 48-hour salt spray test is implemented for the 16 test samples to find out the closed adhesion and optimal test conditions. It is found that Group 9-3134 salt spray test RN 10 has the best adhesion.

This study aims to discover the relationship among different variables, e.g., adhesion of glaze, kiln temperature and firing time, to create and provide steadier, more convenient and easily operated “glaze” firing method to increase the application existing glaze. Generally, the enamel is applied to copper, iron, gold and silver base. The material melting point is about 800 ℃ and delicate surface can be produced.

The optimal combination of copper plate and glaze sintering process is obtained by Taguchi method. The properties and manufacturing technology of copper plate and glaze are used to control the quality. One-way ANOVA is used to obtain the p-value. Sintering temperature is positively correlated to RN, which denotes that higher sintering temperature leads to stronger sintering adhesiveness.

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