FACTORS AFFECTING THE STABILITY OF ENAMEL COATING ON STEEL

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ABSTRACT:

Vitreous enamel coating is a protective coating for metals to give a tightly adherent hard finish resistant to many abrasive and corrosive materials. For these reasons it is widely used in many applications such as domestic cookers, stoves, refrigerators, heaters etc.
In the present work, an attempt has been made to study some parameters such as drying time, firing temperature, quartz additions, acid concentration and Nickel deposition on stability of enamel coating on steel sheet in both acidic and basic medium.
Results show that increasing either soaking time, drying time decreases loss in weight. The suitable firing temperature is found between 750–850 °C that give minimum loss in weight. 1*N* – solution of NaOH at 80 ° C shows higher loss in weight for cover'coat, 6% citric acid has higher attack on samples containing less quartz. Concentration of $NiSO_4$ has no effect on the percentage of loss in weight either for ground or cover coat.

KEYWORDS: Vitreous enamel, Corrosion Resistance, Ceramics, Enamel Coating, Surface Treatment, Protective Coating.

INTRODUCTION

Many studies were done for enamel coating on steel sheet [1-4]. Special grade steel was used in order to ensure good coating adhesion in direct vitreous enamelling [5]. The surface of which is first pickled in sulphuric or phosphoric acid and then plated with nickel, before application of enamel. Enamel coating is effective in the protection of corrosion-resistance steel surface from oxidation during heating [6]. A number of cast steels coated with enamels EVT-100 were hold at a temperature 800 – 1250 °C. Effect of heating on oxidation-resistance, mechanical properties, corrosion-resistance, steel microstructure were studied also liquid technique of boron containing layer forming on a steel surface is considered [7]. Boronizing is studied as a surface pretreatment technique for groundless enamel deposition. Liquid boronizing was found to decrease steel oxidation at initial stage of baking. Gas evolution from the steel is also decrease. The support coating with enamel can be used in automobile industry (voltage regulators), rocket, and space techniques as well as in medical [8] and household ware [9].
In this paper the factors influencing the stability of enamel coating on steel sheet were studied and the results of the experiments were represented and discussed.

MATERIALS AND EXPERIMENTAL

MATERIALS:

The raw materials of enamel coating are inorganic materials that available in nature like quartz, clay, and feldspar. The raw materials are divided into two categories:

A– FRIT:

This is the main constituent of enamel, it consist of different oxides which reduce the softening point when the sample is fired in furnace. Table 1 and 2 show different frit compositions that used for ground and cover coat:

Table (1): Frit Composition for ground Coat

Compound	Composition
Quartz	10
Feldspar	30
Borax	40
Sodium nitrate	3
Sodium carbonate	15
CaO	0.4
NiO	0.85
CoO	0.05
CaF_2	0.7

Table (2): Frit Composition for Cover Coat

Compound	Composition
Quartz	35
Borax	38
TiO_2	14
ZnO	3
Al_2O_3	1
KNO_3	6
CaO	0.04

B – BALL-MILL ADDITIONS:

The batch of the ball-mill addition which consists of 100 gm frit and other materials has the following composition.

Table (3): Ball-Mill additions forming ground coat

Compound	Parts (gm)
Frit	100
Quartz	18
White Clay	8
Blue Clay	2
Borax	0.4
Sodium nitrate	0.2
Magnesium Carbonate	0.1
Water	50

Table (4): Ball-Mill additions forming cover coat

Compound	Parts (gms)
Frit	100
Blue Clay	4
Sodium aluminate	0.3
Potassium Carbonate	0.2
Water	40

APPLICATION OF THE ENAMEL:

After drying of steel sheet specimens the steel is ready for enamel coating, the granules of the frit are grounded together with water and mill additions the grinding is carried out until 3-6% solid remains on a 200-mesh screen. The obtained slip from the ball mill aged 24 hrs before use. The steel specimens are dipped in the slip so a thin layer of it is formed on the surface of the specimens. The formed layer is dried rapidly in drying oven at a temp of 120–150 °C. The specimens are fired in a muffle furnace for about 5 min at 830–850 °C. The composition of the frit and ball mill addition for ground coat is shown in tables 1,3[10].

COVER COAT APPLICATION:

The grinding is carried out up to 3–6 % solids remain on 200 mesh screen and the same procedure applied as in ground coat. The formed layer of cover coat is dried in ordinary dryer at temperature of 120–150 °C. The drying process followed by firing at 800–830 °C for 5 min. The compositions of the frit and ball mill addition for cover coat are shown in tables 2, 4.

PREPARATION OF METAL FOR ENAMELLING:

Pretreatment of steel structure prior to enameling consists of:

a- removal of any grease or oil residues using alkaline cleaners of different concentration, but in general they are between 25–40g/l and at operating temperatures 80–95 °C.

b- Removal of any rust by using acid pretreatment. Usually cold HCL with concentration 10% is still in use.

c– Neutralizing.

The last step in cleaning sequence must be alkaline to ensure that all acid solution is neutralized. Usually a simple dilute solution of hydrated borax and soda ash at 70 – 75 °C was used. The strength of neutralizer need not to exceed 0.1%.

d– Rinsing.

Effective rinsing between each stage of the metal cleaning process is essential.

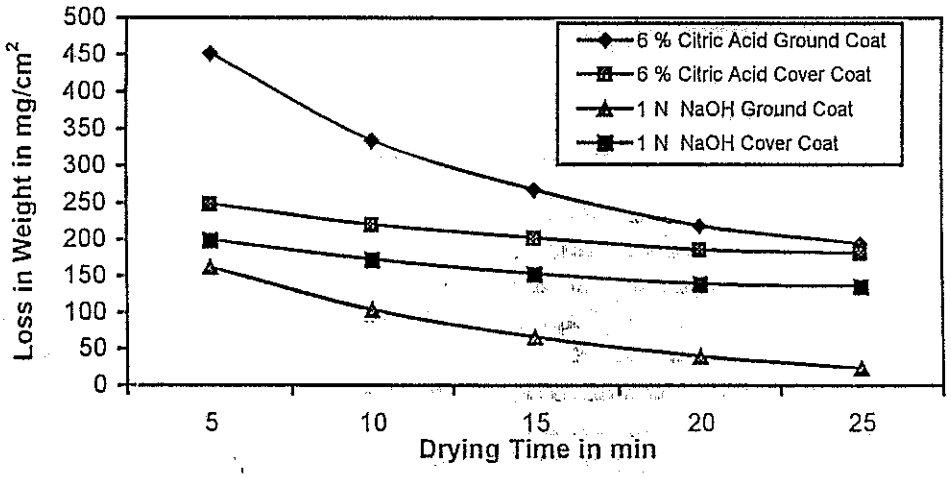

Figure1: The Effect of Drying Time on both Base and Acid resistance of enamel

RESULTS AND DISCUSSIONS:

1- Effect of Drying Time:

Figure (1) shows the effect of drying time on loss in weight for acid resistance (6% citric acid at 128 °C for ground coat and cover coat and for base resistance (1N NaOH, 80 °C) for ground coat and cover coat. Results showed higher attack with acid especially for ground coat than for cover coat. This was attributed to the higher percent of alkalis present in the ground coat composition, that react with acid medium and causes higher loss in weight. As the drying time increase loss in weight decreases due to increasing drying time, the behavior can be explained as follows: At short drying time, the amount of remaining water in the enamel layer after the drying process is high. When the drying specimen enters the firing process, water evaporated rapidly and it form pores and cavities within the enamel surface. These pores and

cavities. Allow the chemicals to diffuse and attack the enamel surface either ground coat or cover coat, this in turn will decrease the adherence between the enamel layer and steel sheet.

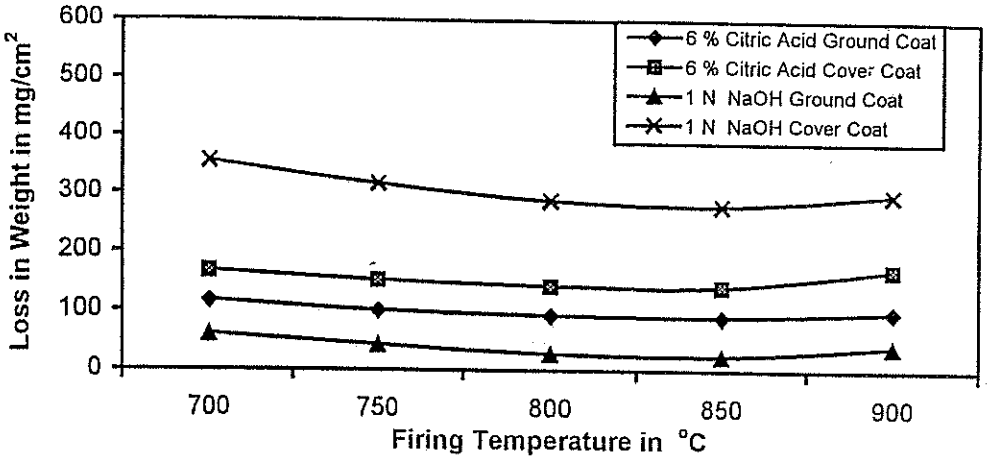

Figure 2: The Effect of Firing Temperature on both Base and Acid resistance of enamel

2-Effect of Firing Temperature:

Figure (2) shows the effect of firing temperature on loss in weight for acid and alkaline medium. From figure it was found that there is an optimum firing temperature between 750 and 850 ° C, which agrees with that found in literature [11].

The behavior of the enamel layer can be explained as follows: at low firing temperature, some reactions between the enamel components and enamel/steel interface are not carried out so, the glassy phase will not be completely formed. There is an optimum temperature for the glassy phase to be formed, which is more resistance for both acid and alkaline mediums as shown in Figure (2).

3- Effect of Acid Concentration:

The increase of the acid concentration used in pickling leads to increase the adherence between the enamel layer and the steel sheet that leads to increase the amount of rust removed from the steel sheet surface [12, 13].

From Figure (3) it can be said that in case of ground coating changing of the H_2SO_4 concentration from 5% to 15% (pickling solution) has no effect on the acid resistance of enamel surface but above 20% H_2SO_4 concentration, the acid resistance decreases suddenly. While in case of cover coat no effect on the acid resistance for all values of H_2SO_4 concentration.

This can be attributed due to the fact that at high concentration of H_2SO_4, the acid roughen the surface of the steel sheet with forming pores and cavities, the enamel layer between these pores is very thin leading to easy attack either from citric acid or 1N NaOH at 80 °C. The losses 1N-NaOH are much higher for the cover coat than for ground coat.

4- Effect of Quartz Addition:

Figure (4) shows relation between the quartz addition and acid and base resistance. It can be seen from the figure that the increase of the quartz addition leads to increase the acid resistance of the ground coat while there is no effect on the

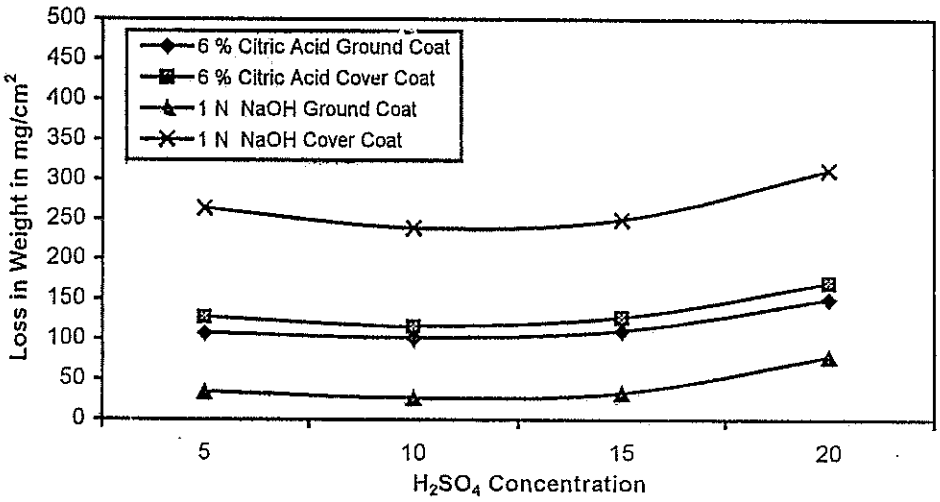

Figure 3: The Effect of Sulphuric Acid Concentration on both Base and Acid resistance of enamel

cover coat. Due the difference in composition of cover and ground coat, it can be seen from Figure 4 that with increasing of quartz percent in ground coat the loss in weight is increased while in case of cover coat there is no remarkable effect.

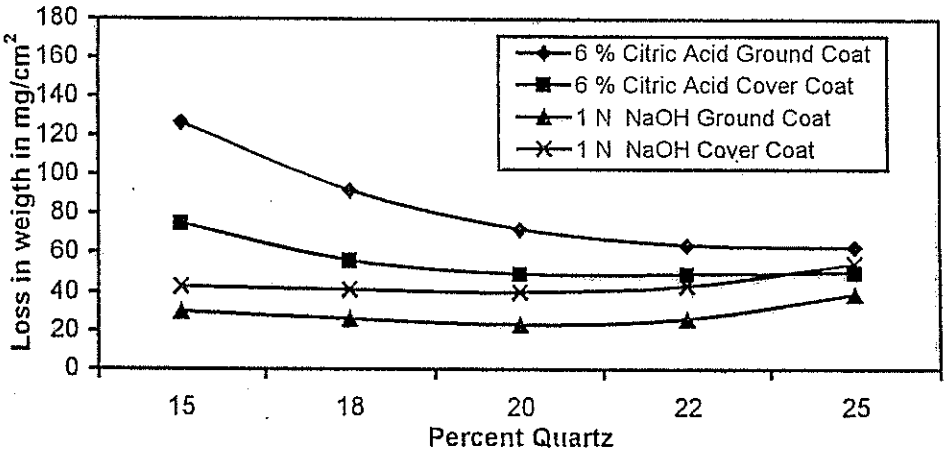

Figure 4: The effect of the Quartz Addition on both Base and Acid resistance of enamel

5- Effect of Nickel Sulphate Solution:

Figure (5) show the relation between $NiSO_4$ concentration and the acid resistance and base resistance for ground coat and cover coat. It can be remarked from the figure that $NiSO_4$ concentration has no effect on the acid resistance or base resistance for both ground and cover coat. This can be attributed to that nickel ions as transition element is deposited on the surface of steel sheet and increase the adherence so the losses of weight resulted from the impact test decreased and do not affect the properties of the produced enamel surface [14].

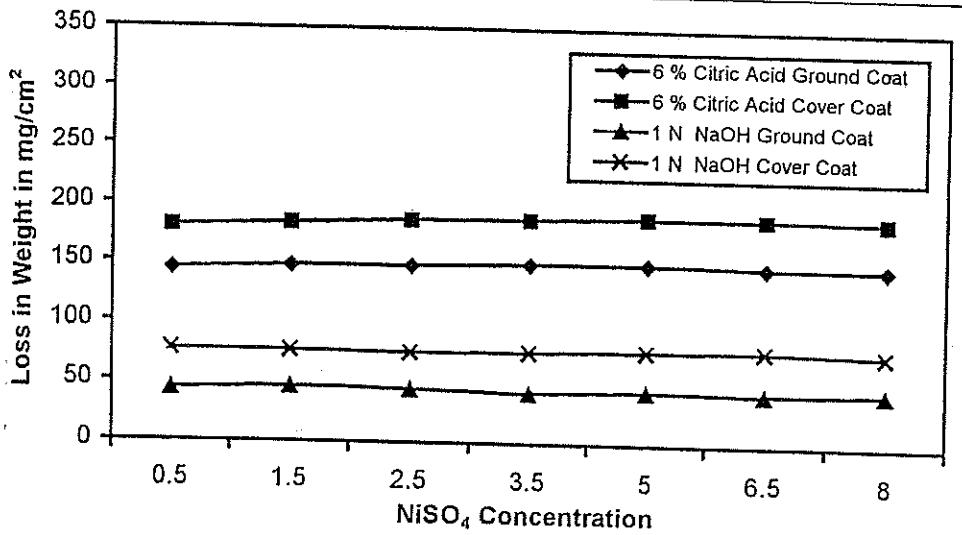

Figure 5: The Effect of the $NiSO_4$ Concentration on both Base and Acid resistance of enamel

CONCLUSIONS:

Increasing the soaking period increases both the acid and base resistance. The drying time should be not less than 10 min to prevent the macropores formation during firing process which can decrease the enamel/metal adhesion force as well as the acids and base resistance.

Increasing the firing temperature increases the enamel metal adhesion force the best surface quality of enamel can be obtained at the firing temperature of 850 °C after the acids and base resistance tests.

Increasing the pickling acid concentration increase the adherence between the enamel layer and steel sheet until reaching the optimum condition at acid concentration of 10%. The acid and base resistance tests of the enamel surface showing that the acid concentration of 20% should be avoided.

Increasing $NiSO_4$ concentration lead to increase the adhesion force in ground coat/metal interface, the increase of $NiSO_4$ concentration has not any influence on the other properties of enamel surface.

Increasing the quartz percent decrease the enamel/ metal adhesion force as well as the base resistance of the ground coat. Increasing the quartz percent increase the acid resistance of the ground coat.

REFERENCES:

1. Y. Mosttefai, M. Auriac, M. Shanahan, J. Bressan, A. Meslif, International J. of Adhesion and Adhesives, Vol. 18, No. 4, pp. 273-281, (1998).
2. G. Ernie, K. Pratap, European Coatings Journal, No. 5, pp. 342-349, (1998).
3. A.P. Zubekhin, N.V. Gurnovich, A.V. Guzir, Glass and Ceramics, Vol. 52, No. 11-12, pp. 316-318, (1995).
4. J. H. Cheland, P.G. Morgan, Engineering Failure Analysis, Vol. 3, No. 3, pp. 149-155, (1996).
5. H. Warnke, Galvanotchnik, Vol. 85, No. 7, pp. 2167-2175, (1994).
6. A. G. Bratukhin, Metallovedenie-I-Termicheskaya-Obrabotka-Metallov, No. 12, pp.18-21, (1992).
7. A.P. Zubeekhin , N.V. Gurnovich, A.V. Guzij, Steklo-I-Keramika, No. 11, pp. 25-27, (1995).
8. N.M. Bobkova, Khimiya-I-Technologiya-Topliv-o-Masel, No. 4, pp. 5-9, (1995).

9. N.V. Gurnovich, A.P. Zubekhin, Steklo-I-Keramika, No. 9-100, pp. 35-36, (1994).
10. F. H. Norton, "Element of ceramics", 2nd ed. Addison-Wesley Pub. London, 1974.
11. K. A. Maskall, and White, "Vitreous Enameling "university of Leeds, UK, 1975.
12. T. Farkas, "Corrosion week", Akademia Kiado, aspect, 1970.
13. Hans Kyri, "Bayer wnamel hand book" Vol. 1, 1975.
14. A. T. El-Mallah, I. H. Aly, et al. Metal Finishing, Vol. 91 (5), PP. 19-21, 1993.