

Wear Resistance of a New Glass Ceramic Coating

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ABSTRACT

A new wear resistance glass-ceramic coating system iron (low alloyed low carbon steel) based substrate was developed. The effects of heat treatment conditions and mill additions on wear resistance of developed coatings resistance were studied. The coating materials showed excellent properties for protection the iron substrate from wear. Also, in this work mathematical modeling is implemented and regression equations are obtained by using (SPSS) software to predict the experimental data for wear rate. Comparing the predicted and measured values gives high prediction accuracy.

Keywords: glass-ceramic, wear rate, SPSS software.

مقاومة البلى لطلاء سيراميك زجاجي جديد

الخلاصة

في هذا البحث تم تطوير طلاء سيراميك زجاجي جديد مقاوم للبلى لمعدن الحديد (فولاذ منخفض السبك منخفض الكربون) كمادة اساس. تم دراسة متغيرات المعاملة الحرارية زاضافات الطحن على مقاومة البلى للطلاء المطورة. ابدى الطلاء المستخدم خواص حماية ممتازة لمادة الاساس الحديد ضد البلى. كذلك في هذا البحث تم عمل موديل رياضي وتم الحصول على معادلات انحدار باستخدام برنامج (SPSS) للتنبأ بالبيانات العملية لمعدل الطلاء. اعطت مقارنة النتائج المقاسة بالنتائج المتنبئة دقة تنبأ عالية.

INTRODUCTION

One of the main reasons for shutting down of units like thermal power station, jute/textile mills, slurry transportation systems, etc. is the high wear rates of their components. Minimized wear would not only contribute directly to the

reduction of unit cost of components but also downtime reduction [1].

Wear, in general, is a gradual digressive process, whereas fracture occurs as a sudden failure. Thus wear in general may be considered as damage to a solid surface involving progressive loss of materials either due to sliding effect with another contacting surface or due to chemical attack in corrosive/high temperature environment. Depending on its nature, the wear can be one of the following four types viz. adhesive, abrasive, erosive and corrosive [2].

The strategy to reduce wear is to first change the material of the component to those that have higher hardness, higher strength and long survivals. However, the option is not always feasible because of the following reasons: (i) it is not cost-effective in many situations and (ii) the concerned component is so complicated in shape and design that technically it is not feasible to change the material. The other strategy will be to modify the surface so that it has higher hardness. This can be done through surface diffusion of materials or alternately by applying a coating on the surface. The coating can be of a polymer or of a glass-ceramic. The advantage of a glass-ceramic coating is that it is chemically inert, can withstand high temperature (up to ~1000°C) and has superior mechanical properties compared to polymer and other non-oxide coatings, viz. paints, metals, rubbers, etc. Polymers can generally withstand a temperature of ~ 250°C and in rare instances, it can go up to 400°C [2]. A glass ceramic coating often allows more flexibility in design as it can withstand much wider mechanical and thermal abuses. This paper describes the preparation, application, and evaluation of a new wear resistance glass-ceramic coating which can be applied directly on steel panels. These new coatings are designed for application on various grades of steel. The effects of quartz addition and crystallization treatment on properties of the ceramic coating have been studied. Mathematical model for these effects was also created.

Experimental work

Sample preparation

The samples used as substrate for enamel coating were rectangular plates (20mm×30mm with a thickness of 1.5mm) of low alloyed low carbon steel, their chemical composition is shown in Table (1). The sample surface is exposed to a jet of an abrasive material shot to remove scale, rust and dirt. The surface becomes clean and slightly pitted which helps promote good chemical and physical bonding. Removal of material from the surface should not be excessive and is controlled by adjusting the air pressure and exposure time of blasting.

Before blasting, oil and drawing compounds are removed by heating the surface at 455°C to burn off the organic contaminants.

Table (1) Chemical composition of metal substrates

Element	C	Mn	Ni	Al	Co	Nb	W	Fe	Si	Cr	Mo	Cu	Ti	V	Pb
Low carbon low alloyed steel (%wt)	0.18	0.12	1.35	0.05	0.05	0.05	1	94	0.05	1.3	0.4	<0.05	<0.05	<0.05	<0.05

Frit manufacturing

Glass frit is the major constituent in bisque (unfired) enamel coating. Frit is the homogeneous melted mixture of inorganic materials that is used in enameling steel process. Frit is prepared by fusing a variety of minerals in a furnace and then rapidly quenched the molten material.

In this study we developed coating material which is designing for application as a single-coat on various grades of steel alloys.

The frit was prepared from reagent grade chemicals: SiO₂, Al₂O₃, Na₂B₄O₇.10H₂O, CaCO₃, Na₂CO₃, ZnO, P₂O₅, TiO₂, Li₂CO₃, CoO, NiO and CaF₂. The batch was evenly blended and melted in a graphite crucible in resistance furnace at 1250°C, the batch hold at this temperature for 2 hr in order to the all of the raw materials reacted and the batch become a homogeneous, bubble-free liquid. Then a stream of the smelted and refined molten batch is drawn from the graphite crucible and quenched into water to produce a coarse granular frit.

The coarse frit was milled by using a ball mill with alumina ball as the grinding media and screened with a mesh size of 200 mesh, stored in an oven at 100°C to prevent problem with moisture. The chemical composition of the frit powders is listed in Table (2).

Table (2) Chemical composition and weight percent of frit

Compound	SiO ₂	Al ₂ O ₃	B ₂ O ₃	Na ₂ O	P ₂ O ₃	Zn O	TiO ₂	CoO	NiO	CaO	Li ₂ O	F ₂
% (wt)	52-55	0.5-2	12-16	6-9	3-5	2-4	1-5	1-2	0.5-1	2.5-4	2-5	1-3

Preparation of enamel slip

For application of the coating, the frit is further processed by mixing it with certain mill-additive to make thick slurry called "slip" to enable its uniform and thin application on the clean metal parts by dipping technique.

The rheological properties of coating material slips are very important for ensuring proper application of the coating on the substrate. Kaolinite, borax, and water were

added to form a batch of enamel slip. Refractory materials can be added to impart desired properties to the fired coating. In this work quartz was added in different amounts (5,10,15%) to study their effect on coating properties. The specific gravity of the enameling slip was measured using an electronic weighing balance and was controlled between (1.7-1.8) by adjusting the water content. Subsequently, the slip was aged for 24 hours before enameling to improve its fluidity. Table 3 shows the weight percent composition of enamel slip.

Table (3) Weight percent composition of coat slip

Compound	Frit	Clay	Borax	Quartz	Water
Weight (wt%)	100	7	1	0,5,10,15	50

Coating application

The coating material in the slip form is applied on the clean metal surface by dipping method. Dipping method is a simple and quick technique requiring no special plant, where the specimen is immersed in the enamel slip, withdrawn, allowed to drain.

After application of coating slip, the coated samples were dried in an oven at 120°C for 15min to remove moisture. The dried coated samples are then fired in a box furnace at 860°C for 10min, the coating material then fuses and reacts with the clean metal surface to form a strongly adherent coating. Subsequently, to obtaining the glass-ceramic coating, the enameled samples were held in a furnace at different temperatures (500, 550,600)°C for 60 & 120 min, respectively. The entire process of glass-ceramic coating is summarized in the flow diagram of experimental program as shown in Fig 1. Coated sample is shown in Figure (2).

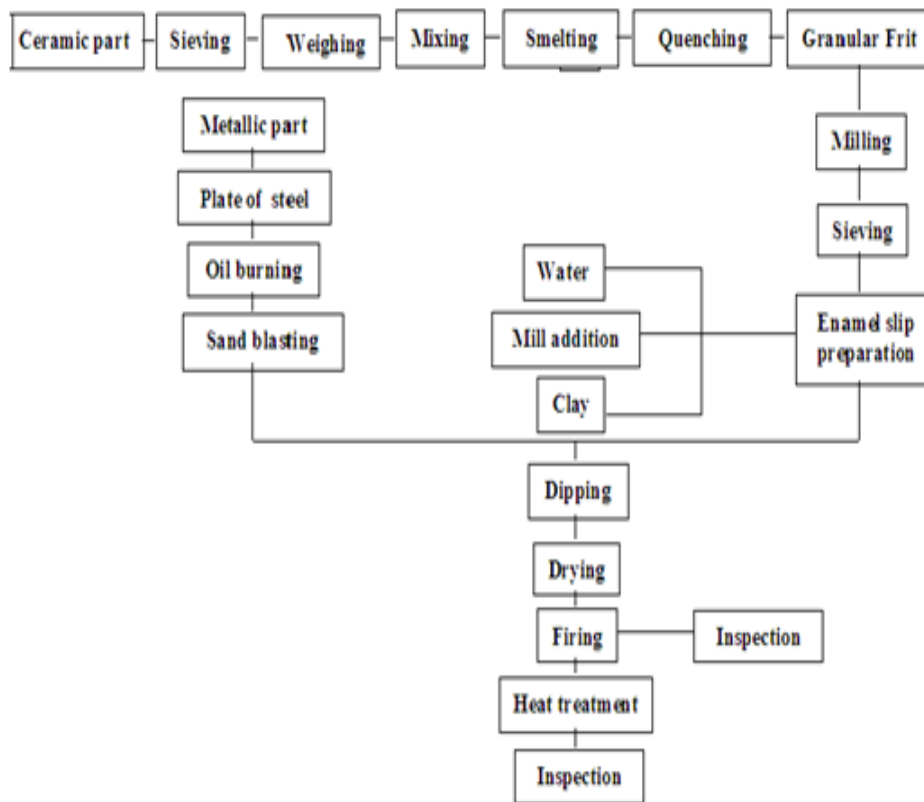


Figure (1) Flow diagram of experimental procedure

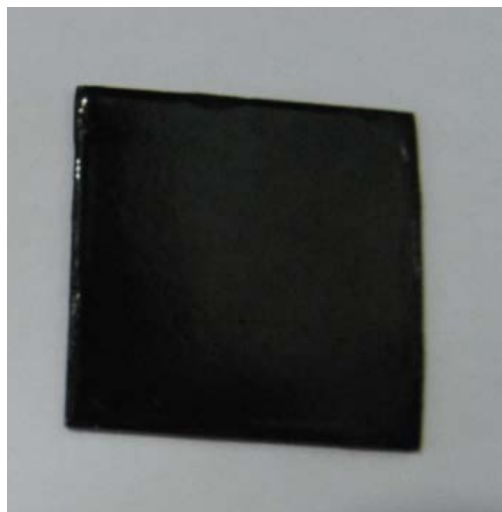


Figure (2) Coated sampl.

**Inspection and testing
Coating characterization**

The thickness of the resultant coating was measured by an eddy current based thickness measuring instrument with ND-2 type probe, suitable for non-ferrous alloys. Phase analysis of the resultant coating before or after heat treatment was done by X-ray diffraction analysis (Philips PLO1840 X-ray diffractometer in 2θ ranging between 10° to 90° using Cu $K\alpha$ radiation[3]. Figure (3) shows the X-ray diffraction patterns.

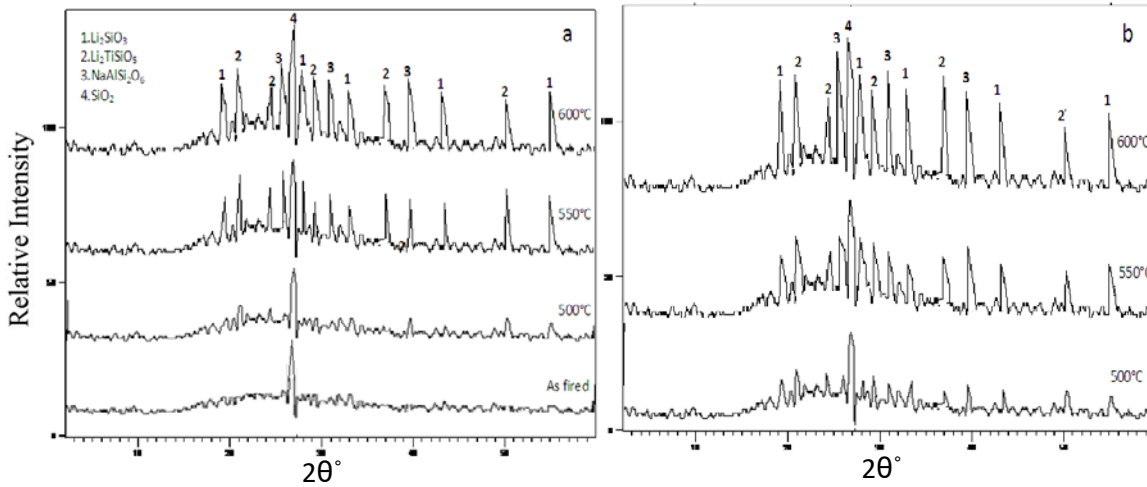


Figure (3) XRD patterns of samples (group D) for different temperatures at two times a: 60 min, b: 120 min [3]

Adherence strength test

In accordance with EN10209,[8] the test in Europe standard of enamel adherence strength, the enameling specimen is tested by the steel ball falling impact. Following the destruction, the adherence strength was judged according the relics of enamel on the destroyed surface. The enamel adherence strength can be graded into 1st, 2nd, 3rd, 4th and 5th grade and the 1st grade is the best. If the most of the enamel layer is removed from the steel sheet, and the surface appears silvery bright after the impact, the adherence strength is poor and 5th grade. If the most of enamel layer remains on the steel sheet, the adherence strength is excellent and 1st. The results show the coated samples have good adherence strength. The results were discussed in [4]. Figure (4) shows the peeling surface of coating with adherence test.

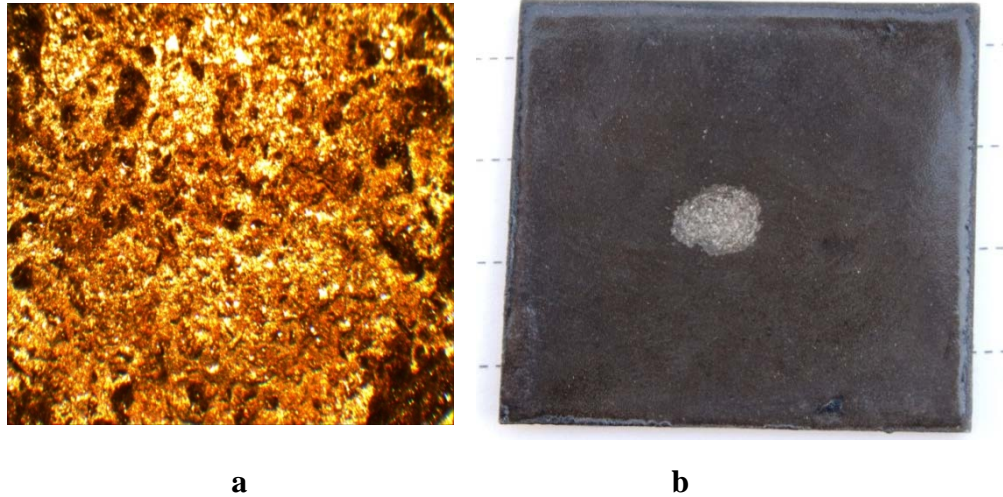


Figure (4) Peeling surface of coating with adhesion test. a. microstructure (200x) peeling surface. b. photographs of peeling surface.

Microhardness measurement

The hardness of the specimens was measured with a Vickers micro-hardometer model (HVS-1000). The hardness tests were performed under an indentation load of 50 g for 20 s. In order to obtain reliable statistical data, analysis points were spaced so as to eliminate the effect of neighboring indentations, and the hardness was evaluated by taking five indentations on each specimen and averaging of only three middle values[4].

Wear test

Wear tests were performed in accord with the ASTM G99 for wear testing with a pin-disk apparatus. The wear rate of the materials were determine at a load of 10N, rotating speed of 2cm/sec, sliding distance of 50m using a pin-on-disk tribometer. The wear rate expressed in (mg/cm) is calculated as follows:

$$W.R = \frac{\Delta w}{S} \quad \dots \dots \mathbf{1}$$

Where: *W.R*: wear rate, Δw : weight loss in (mg), *S*: sliding distance in (m).

All of the tests were conducted at ambient atmospheric condition at room temperature (25)°C. Lubrication is not applied to avoid the complication of terbo-chemical effects.

Mathematical modeling

A statistical model for the prediction of the wear rate of the resultant coating was created by regression function in SPSS software from the training data set. The definition of the variation factors (independent variables) and their values are given in Table 4, while the dependent variables (response functions) were wear rate (W.R). All original 28 samples within experimental data shown in Table 5 were randomly divided into two data sets; the training data set and the testing data set. The training data set contained 20 samples which were used to build a prediction model as shown in Table (6) and the testing data set contained 8 samples which were used to test the flexibility and the validity of the prediction model as shown in Table (7).

Table (4) Definition and values of independent variables used in regression equation.

Designations of independent variable	Name of variable	Value
X_1	quartz addition (% wt)	0,5,10,15
X_2	holding time (min)	0.60,120
X_3	temperature of heat treatment (°C)	0,500,550,600

Table (5) Original experimental data for predicted and measured wear rate.

No	Quartz added (%)	Temp.(C°)	Time (min.)	Measured wear rate (mg/cm)	Predicted wear rate (mg/cm)
1	0.00	0.00	0.00	2.415	2.41332
2	0.00	500.00	60.00	2.368	2.40195
3	0.00	500.00	120.00	2.234	2.17906
4	0.00	550.00	60.00	2.183	2.15647
5	0.00	550.00	120.00	1.847	1.91391
6	0.00	600.00	60.00	1.881	1.86641
7	0.00	600.00	120.00	1.614	1.60418
8	5.00	0.00	0.00	2.409	2.41301
9	5.00	500.00	60.00	2.359	2.38048
10	5.00	500.00	120.00	2.221	2.17742
11	5.00	550.00	60.00	2.169	2.13091
12	5.00	550.00	120.00	1.833	1.90818
13	5.00	600.00	60.00	1.797	1.83675
14	5.00	600.00	120.00	1.633	1.59435

15	10.00	0.00	0.00	2.411	2.41028
16	10.00	500.00	60.00	2.336	2.35659
17	10.00	500.00	120.00	2.198	2.17335
18	10.00	550.00	60.00	2.181	2.10292
19	10.00	550.00	120.00	1.822	1.90001
20	10.00	600.00	60.00	1.775	1.80466
21	10.00	600.00	120.00	1.627	1.58209
22	15.00	0.00	0.00	2.406	2.40511
23	15.00	500.00	60.00	2.311	2.33027
24	15.00	500.00	120.00	2.187	2.16686
25	15.00	550.00	60.00	2.132	2.0725
26	15.00	550.00	120.00	1.820	1.88942
27	15.00	600.00	60.00	1.719	1.77014
28	15.00	600.00	120.00	1.620	1.5674

Table (6) Training data for wear rate.

No	Quartz added (%)	Temp.(C°)	Time (min.)	Measured wear rate (mg/cm)	Predicted wear rate (mg/cm)
1	0.00	0.00	0.00	2.415	2.41332
2	0.00	500.00	60.00	2.368	2.40195
3	0.00	500.00	120.00	2.234	2.17906
4	0.00	600.00	120.00	1.614	1.60418
5	5.00	0.00	0.00	2.409	2.41301
6	5.00	550.00	60.00	2.169	2.13091
7	5.00	550.00	120.00	1.833	1.90818
8	5.00	600.00	60.00	1.797	1.83675
9	10.00	500.00	60.00	2.336	2.35659
10	10.00	500.00	120.00	2.198	2.17335
11	10.00	550.00	60.00	2.181	2.10292
12	10.00	550.00	120.00	1.822	1.90001
13	10.00	600.00	60.00	1.775	1.80466
14	10.00	600.00	120.00	1.627	1.58209
15	15.00	0.00	0.00	2.406	2.40511
16	15.00	500.00	120.00	2.187	2.16686
17	15.00	550.00	60.00	2.132	2.0725
18	15.00	550.00	120.00	1.820	1.88942
19	15.00	600.00	60.00	1.719	1.77014
20	15.00	600.00	120.00	1.620	1.5674

Table (7) Testing data for wear rate.

No	Quartz added (%)	Temp.(C°)	Time (min.)	Measured wear rate (mg/cm)	Predicted wear rate (mg/cm)
1	0.00	550.00	60.00	2.183	2.15647
2	0.00	550.00	120.00	1.847	1.91391
3	0.00	600.00	60.00	1.881	1.86641
4	5.00	500.00	60.00	2.359	2.38048
5	5.00	500.00	120.00	2.221	2.17742
6	5.00	600.00	120.00	1.633	1.59435
7	10.00	0.00	0.00	2.411	2.41028
8	15.00	500.00	60.00	2.311	2.33027

Results and discussion

Wear resistance

The results of wear rate of enamel coating varied from 2.4×10^{-4} mg/cm (for as-fired enamel) to 1.6×10^{-4} mg/cm (for heat treated enamel coating at 600°C for 2hr).

The results showed that there were not significant differences in wear rates between enamel coatings with addition of quartz. In the other hand, the wear rates of as-fired and heat treated enamel coatings are diverse. The difference is attributed to the variation of crystalline phases content. The results present in Fig 5 indicate that the wear rate of heat treated enamel coating improves with increasing both temperature and time of heat treatment. The presence of a certain amount of dispersed crystal phases throughout the coating results in high improvement of wear rate. The area fraction of crystallites in the glassy matrix is expected to enhance the reinforcement to the glass matrix which should lead to the enhancement of wear resistance of the coatings. In literature, Clelland et al. [8] suggested that variation in ceramic composition and microstructure may affect the opposing enamel wear.

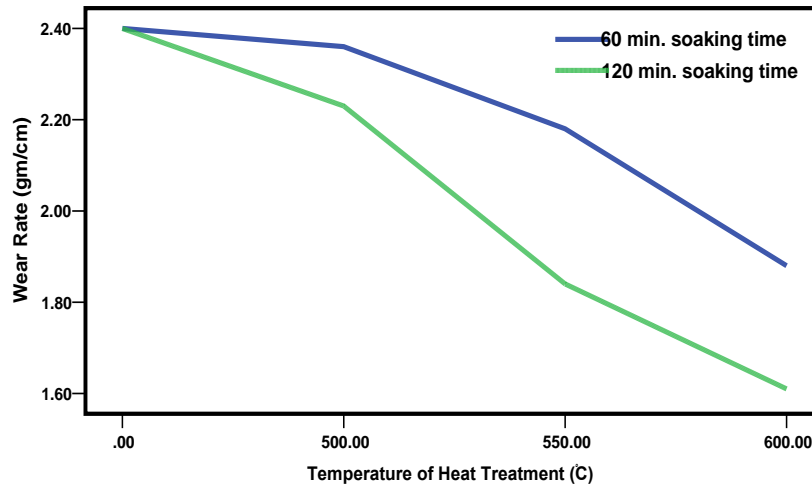


Figure (5) The effect of temperature and time of heat treatment on wear rate of enamel coating.

The results of this study suggest that there is an inverse relationship between the hardness and wear rate. The harder materials revealed more wear resistance as shown in Fig 6. Although similar results have been reported by Borgioli et al.[9], several studies have found no correlation between hardness and wear due to the complexity of wear process [10-13].

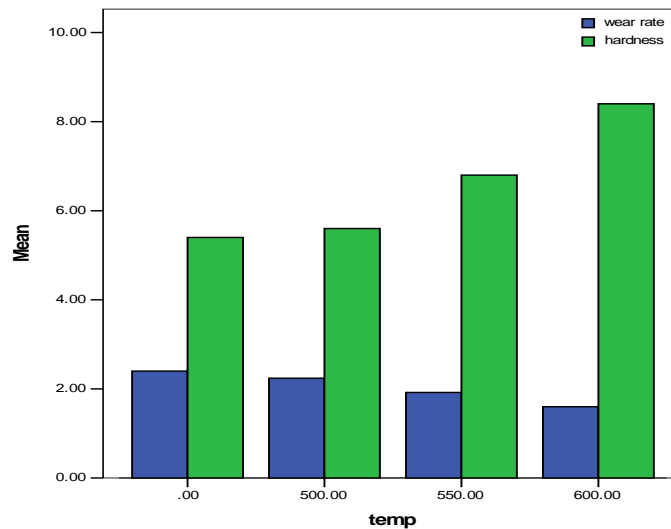


Figure (6) Microhardness (HV) and wear rate of 15%quartz added enamel coating treated with 2hr soaking time.

Mathematical model

After processing of experimental results, mathematical model(regression equation) for wear rate was obtained:-

$$W.R = 2.413 + 0.005X_2 - 4.9 * 10^{-5}X_1^2 - 1.6 * 10^{-5}X_1X_2 + 6.61 * 10^{-5}X_1X_3 - 8.9 * 10^{-6}X_2^2 - 6.6 * 10^{-6}X_2X_3 - 2.4 * 10^{-6}X_3^2 \dots 2$$

Where:-

W.R: wear rate (mg/cm), X_1 : quartz addition(%), X_2 : temperature of heat treatment($^{\circ}$ C), X_3 : holding time(min).

The value of the multiple correlation coefficient R, that tell us how strongly the multiple independent variables are related to the dependent variable, was (0.988) for equation (2).

The result of average percentage deviation (Φ) shows that the training data set (m=20) was (2.37%) and the testing data set (m=8) were (1.47%) for wear rate.

This means that the statistical model could predict the corrosion properties with about (97.63%) accuracy of the training data set and approximately (98.53%) accuracy of the testing data set for wear rate of resultant coatings.

Figure (7) shows the comparison between the predicted values and measured values of 28 original data for wear rate of resultant coatings by using (SPSS) software.

It is clear from this figure that the predicted values are in a close match with the measured values for all coatings.

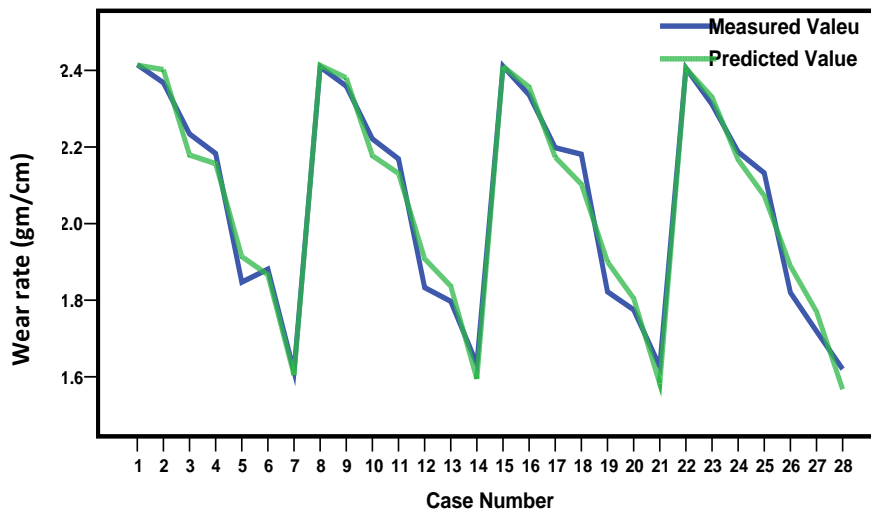


Figure (7) Comparison between Measured and Predicted values for the experimental.

CONCLUSION

The following conclusions may be drawn from the results obtained in this work:

- The coatings were successfully applied as a single coat by simple vitreous enameling process onto already prepared steel surfaces.
- The proposed coating material possesses a reasonable low melting (1200-1250°C) and processing 860°C temperature, leading to less cost and energy consumption during preparation and application.
- Heat treatment improves the wear resistance of the resultant coatings in all cases.
- There are no significant effects of quartz addition on wear rate of resultant coatings.
- The multiple regression models could predict the wear rate with higher accuracy for different quartz addition, and thermal treatment conditions.

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