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Evaluation of Solution Temperature and Treatment Time Effects of Zinc Phosphating on AISI 5140 Alloy Steel

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***Abstract*—**Chemicals coatings are protective surface coatings applied to metals under controlling condition. Phosphating is one of the most widely used metal pretreatment process. Due to its economy, corrosion resistance, wear resistance, adhesion and lubricative properties. The treatment of steel with a dilute solution of phosphoric acid and other chemicals in which the surface of the alloy steel, reacting chemically with the phosphoric acid, is converted to an integral, mildly protective layer of insoluble crystalline phosphate. To achieve better understanding of the coating formation on the alloy steel AISI 5140, zinc phosphate coating process was carried out in lab scale for different phosphating times and treatment temperatures. The influences of immersion time and solution temperature on the surface appearance, coating thickness, coating hardness and roughness were investigated. The results of appearance show defects on the surfaces in conditions of 60, 70°C and phosphate time 1, 2 and 3 min, while good appearance was noted in conditions of 90°C and phosphate time 3 & 4 min. coating weight, coating thickness and coating hardness were increased with an increase of phosphate time and temperature.

***Keywords*—**coatings, corrosion, phosphating, surfaces.

I. INTRODUCTION

The performance of all paints and powder coated finishes depends on the correct preparation of the metal substrates and the failure of coatings in service with problems such as corrosion and blistering are nearly always caused by inadequate pretreatment. The pre-treatment is often more important than the final finish[1]. As well as providing corrosion protection pre-treatment can offer improvements in wear resistance and other valuable properties. The most widely used pretreatment is known as “Phosphating”. This is also defined as a “Conversion Coating”[2, 3]. Corrosion protection, paint adhesion, uniform coverage and cost effectiveness are qualities that have seen sought by metal finishers from the infancy of metallurgy and can all be furnished by the use of a phosphate coating. Phosphating is a relatively simple process that has been used for well over a century to protect metal from corrosion. In general, phosphating is the conversion of a metal surface into an insoluble and integrated lattice of metal and crystalline phosphate. This process takes place by the treatment of the surface with a solution of phosphoric acid and other chemicals, which react with the metal to form a slightly protective layer [4].

The application of the zinc phosphate bath can be carried out at a wide range of temperatures. Although there is no reason to believe that temperatures outside of this range will prevent the composition from having the desirable effects. The temperature of the bath may range from about 60 ºC to 120 °C. The time of treatment of a metal surface with the baths of the various steps need only be long enough to ensure complete wetting of the surface, the contact time of phosphate stage ranges from about 30 sec. to about 300 sec. The broad objective of this

study was to use zinc phosphate coating layer as a base for paint (top coat) to promote good paint adhesion, increase the resistance of the films to humidity and substantially retard the spread of any corrosion that may occurs. So, the aim of this work is to evaluate the effect of treatment time and solution temperature on the zinc phosphating on this alloy. The phosphate layer coat was produced in lab scale using different immersion phosphating temperatures (60, 70, 80, 90 and 100°C) and phosphate time (1, 2, 3 and 4 minutes.) Appearance, coatings weight, thickness, hardness and degree of roughness were evaluated.

II. EXPERIMENTS

Square shape samples of alloy steel (AISI 5140) with dimensions of (45×45×2.4mm) were used Figure 1. According to TT-C-490E and DOD-P-16232[5, 6]. The zinc phosphate coating was applied on the specimens in five successive stages; degreasing, rinsing, pickling, rinsing, phosphating (in different temperature and time), rinsing and drying, as indicated in Figure 2.

20 different conditions were presented in this study, where the zinc phosphating solution temperatures are 60 to 100°C; every condition we increased ten degrees.

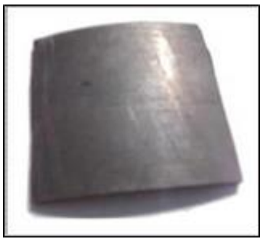

Figure 1. specimen shape which used in experimental part

The phosphating time were carried on between 1 to 4 min as illustrated in Table 1. The p.H value of phosphate solution was 3.2±0.1.

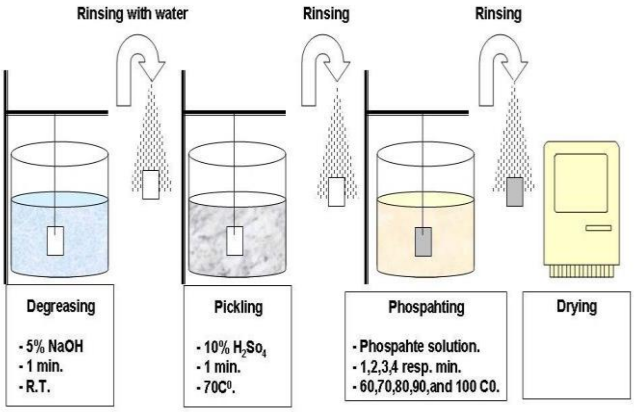

Figure 2. Schematic of zinc phosphating equipment on lab scale shows the steps of process

The specimens were designated below in Table 1 based on the phosphate time, phosphate temperature, and the specimen number. As an example, specimen in the designation **2A1**: **2** indicate the phosphate time (2min), **A** indicates the phosphate temperature (60 ºC), and **1** indicates the specimen number.

Table 1. Specimens Designation.

Gr. NO.	Phosphate time. (min.)	Phosphating Temp. (°C)				
		60	70	80	90	100
1	**1**	1A1 1A2 1A3	1B1 1B2 1B3	1C1 1C2 1C3	1D1 1D2 1D3	1E1 1E2 1E3
2	**2**	2A1 2A2 2A3	2B1 2B2 2B3	2C1 2C2 2C3	1D1 1D2 1D3	1E1 1E2 1E3
3	**3**	3A1 3A2 3A3	3B1 3B2 3B3	3C1 3C2 3C3	3D1 3D2 3D3	3E1 3E2 3E3
4	**4**	4A1 4A2 4A3	4B1 4B2 4B3	4C1 4C2 4C3	4D1 4D2 4D3	4E1 4E2 4E3

Some special tools were prepared for the placement of specimens to providing the proper control of aeration. A 2L glass beaker was used in the process which made from glass to resist the attack by acids. The zinc phosphating bath was controlled by measurement of acidity. This was done using the p.H meter. The bath temperature was maintained at the proper temperature (test temp.), by controlling the heater in conjunction with the beaker. However, depending upon the surface conditions of the base metal, some operations may be omitted or additional operations may be incorporated into the system.

III. RESULTS

A. Visual Inspection

The specified requirements for appearance samples in zinc phosphate coatings are that it be free from gross imperfections, sharp points, and uncoated areas[7, 8]. Also, relatively smooth and uniform in texture and distribution. The resulted photos that filmed for the specimens after the process showed in Figure 3. It's obvious in case of condition one where the specimens treated at solution temperature 60°C for 1, 2, 3 and 4 min respectively, shows poor coatings and white stains on the surfaces. Also, the corrosion products were visible to naked eye as can be seen in Figure 4.

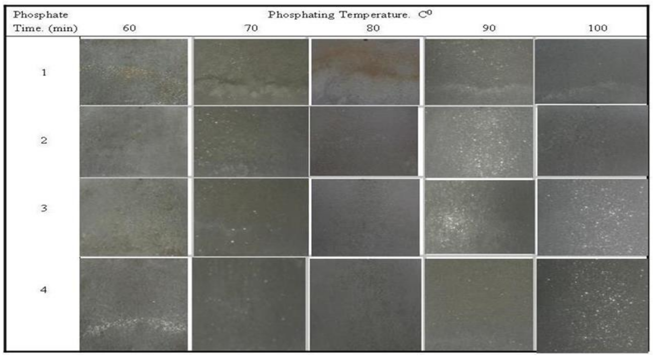

Figure 3. Visual appearance of the surfaces finish after treated in different solution temperatures and for different periods of time.

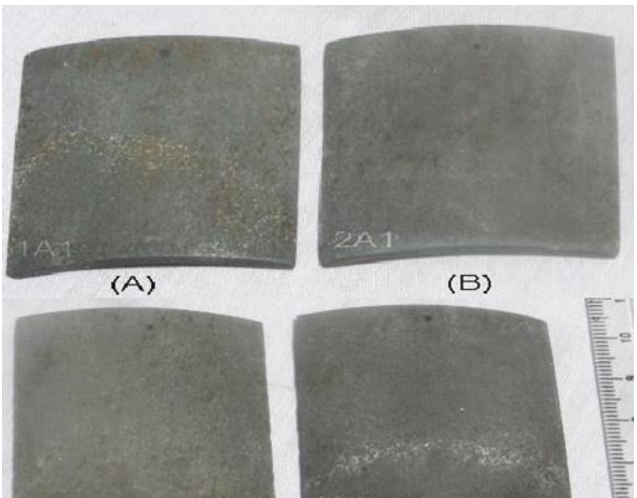

Figure 4 Visual appearance of surfaces after treated in solution temperature 60ºC for different time.

In thic case, the solution temperature was below to the minimum recommended temperature. This low temperature doesn’t enhance the naturalization that occur on the interface between the metal and the solution which effect the formation of the precipitate layer. Vahid et. al. [9] in discussing the effect of immersion time and immersion temperature on the corrosion behavior of zinc phosphate conversion coatings on carbon steel stated that the phosphate coated samples have more positive corrosion potential, lower corrosion current density and higher polarization resistance.

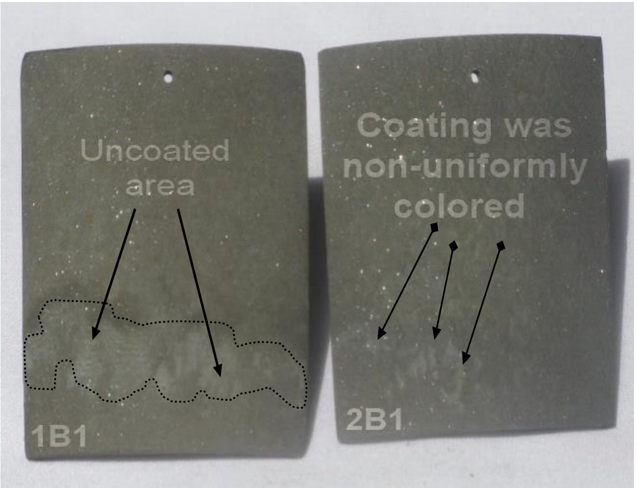

Figure 5. Visual surfaces appearance after treated in solution temperature 70ºC for 1& 2 min.

This indicates that the corrosion resistance of carbon steel improves by the phosphating treatment. Additionally, the anodic and cathodic processes of carbon steel corrosion suppress effectively by the complete coverage of phosphate coatings. He also stated that the phosphate coatings prepared at 45 °C shows the highest polarization resistance and the lowest corrosion current density due to the compact structure of coating. In a study of corrosion behavior of phosphate conversion coatings applied on steel surface, Vahid Asadi, et.al.[9], stated that in low immersion time, the formation of phosphate coating is in the induction stage and incomplete phosphate coating forms. Moreover, the surface is attacked by phosphoric acid and causes the inferior corrosion behavior. Figure 5. shows the surface appearance resulted from samples treated in solution temperature 70^{o}C. Uncoated area, non-uniformly coloured and some of smut where appeared on its surfaces. In addition, (dull luster appearance and low degree of brightness where showed on its surfaces. Figure 6. shows the surface appearance of oxide coatings formed in solution temperature 80^{o}C. The the results were obtained vary from satisfactory appearance to good due to the effect of the phosphating temperature and time, which may have an effect on the rate of reactions. The colour of the zinc phosphate coatings was greyish with bright crystalline spots This is consistent with the observations by Vahid Asadia et. al[9].

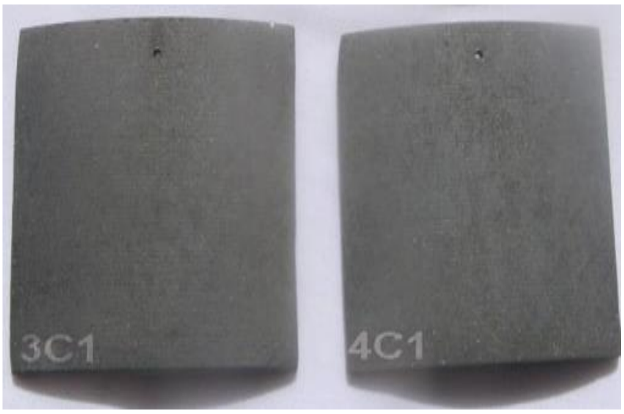

Figure 6. Visual surfaces appearance after treated in solution temperature 80ºC for 3& 4 min.

The surface appearance of the coatings produced in condition of phosphating temperature 90^{o}C is presented in Figure 7. It reveals that the coating mainly has good appearance with varying degree. However, sample which phosphate for 2 min as showed in Figure 7.

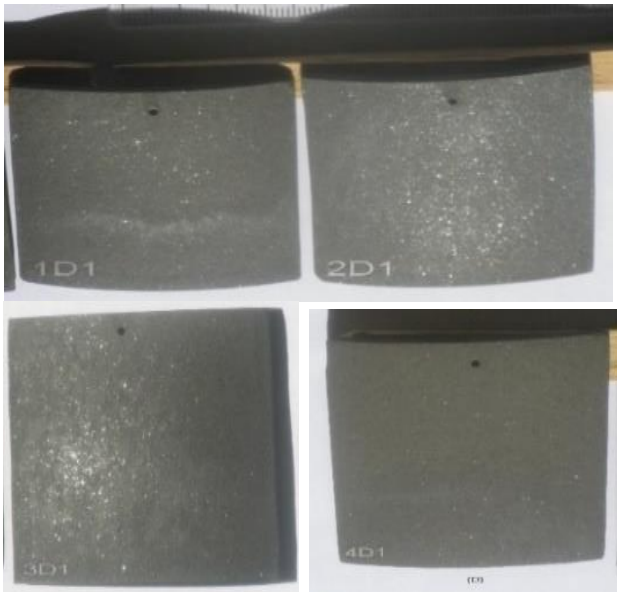

Figure 7. Visual surfaces appearance after treated in solution temperature 90ºC for different time

(1D1) showed a change in color on its surface, because of the lack of good drying after final rinsing stage. In other hand, treatment for 3min has an excellent appearance which means the solution has enough time and higher temperature to react with a base metal (specimen). Therefore, the result was an insoluble layer of phosphate crystals created on the surface. The last condition where the specimens treated at 100^{o}C different time mainly have similar degree to that noted in the appearance of specimens which exposed to phosphate solution temperature 90^{o}C. See Figure 8. The created layers which produced in specimens phosphated for 3 and 4 min (3E1 & 4E1). were uniformly coloured and free of smut, powder, corrosion products, or white stains.

Other references reveal that, other factors can affect on the morphology and corrosion resistance of the coating. H. Eivaz et. al. [10] in discussing the effect of solution temperature and p.H value on morphology of coating on cold steel rolled stated that increasing solution temperature resulted in micro-cracks creation and lack of consistency on the surface of the conversion coating. Moreover, the morphological structure changed with increasing pH value.

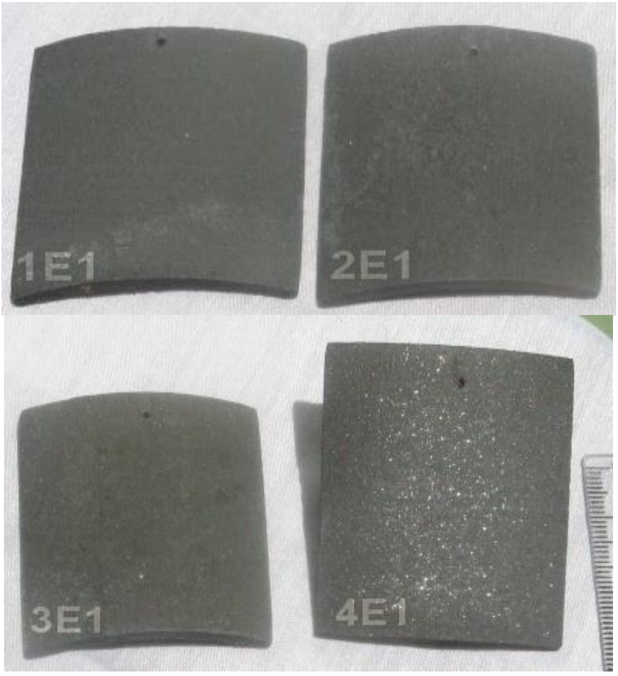

Figure 8. Visual surfaces appearance after treated in solution temperature 100°C for different time.

B. Coating Thickness and roughness

Thickness of the coating is considerable one of the crucial factors in the choice of the performance parameter of surface coating. The thicknesses of the conversion coatings adhering to the substrates produced in different phosphating solutions temperature for different time (Figure 9) were found to be in the range 2.0 to 25.0 μm. The thicknesses of these coatings change depending on the temperature of phosphate solution and the process time. The linear growth of thickness and roughness of the produced coatings with solution temperature increasing has been revealed. Each of the conditions provide a phosphate coating with slightly different properties. This allows a more specialized coating to be selected for the particular application required for part of the structure. The highest coating thickness in one side, 25.0 μm was achieved in condition 4 where the process time was 4 and phosphate temperature was 100°C. The lowest value, 2.1 μm was found for the coating produced in the condition 1. The coatings thicknesses of the samples that phosphated in solution at 60 °C & 70°C are of poorest quality due to nonuniform phosphate coating. As can be seen in Figure 9 the coating thickness on the specimens that phosphated in solution temperature 80°C & 90°C at phosphate time 3 & 4min were the higher thicknesses.

The coatings thicknesses are controlled by the condition of the surface as well as the phosphate time and temperature. The adhesion of phosphate coating to the base metal, as determined by flexing of the metal, varies with the type and thickness of the coating. Generally, heavier coatings are composed of large crystals, which do not bond to each other or to the surface of the metal as well as do fine-grain, thinner coatings [11].

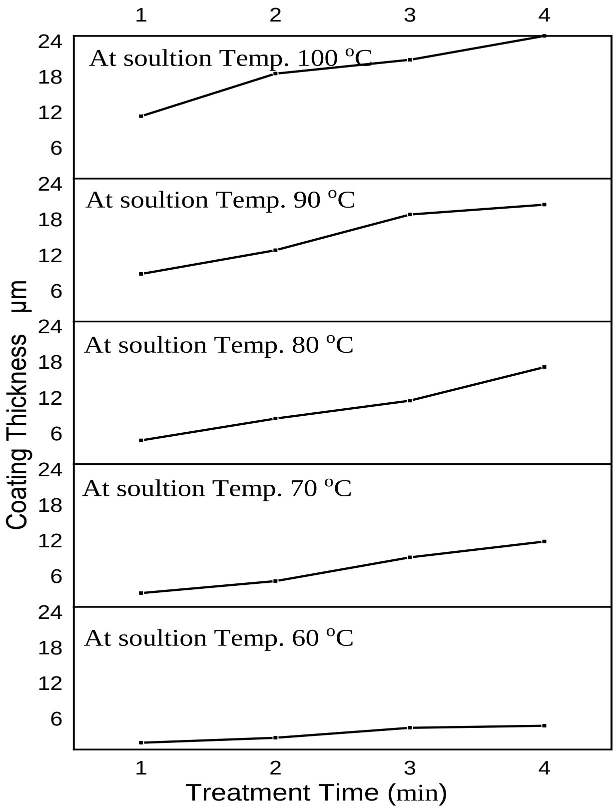

Figure 9. Coating thickness vs. phosphating time at different phosphate solution temperature

Consequently, where adhesion and flexibility may be a problem because of the nature of the application, phosphating material is selected that produces a thin, fine-grain coating[12]. However, this may not result in maximum corrosion resistance. Organic additives and special accelerators added to a zinc phosphate process provides a microcrystalline structure that exhibits optimum paint adhesion and corrosion resistance. Adhesion between the films and the substrates was controlled by pretreatment of the substrates and the thickness of the films, and this is consistent with the results reported by S. Wirasate and F. Boerio[13]. When used as a base for paint films, zinc phosphate coatings promote good paint adhesion, increase the resistance of the films to humidity and water soaking, and substantially retard the spread of any corrosion that may occur. To accomplish this purpose, one of the factors of preparing the metal to be coated should have some micro roughness[14]. Phosphate surfaces enhance this factor, i.e. some degree of roughness help the penetration of the coating into the metal and arise the adhesion of this coat. Figure 10 it’s noted that as increase of phosphate temperature and phosphate time the roughness measurement (Ra) increases. In the absence of a more reliable criterion for evaluation of the roughness measurements of phosphate alloy steel, the adhesion of the outer coat (paint film) will be used for comparison of performance of the tested specimens**.**

C. Coating Hardness

The overall effect of increasing the hardness number value is preferred for providing good wear resistance of metal surface, which is very important for the application of the motor case that will be coated. The hardness testing results that obtained for each group and the effects of time and temperature of phosphating that present in Figure 11. The data obtained showed no high difference in results in same phosphating temperature and time. Figure 3.9 shows the hardness values on produced zinc phosphate layer in the specimens that immersed in phosphate solution at 60^{o}C and at different time are close to the specimen without coat. This may result to an incompletely formed coating due to low phosphate temperature. It is also showing that no obvious difference in values of hardness measurements due to the increasing in phosphating temperature to (70^{o}C). While it shows slight change in hardness reading due the increasing in phosphate temperature to (80^{o}C & 90^{o}C). This resulted from enhancing the rate of reactions that occurred during phosphating process and formation of zinc coat layer on the surface of specimens. In other hand the phosphating temperature arise to 100^{o}C shows the largest values of harness number, which is obvious in phosphate time 3 & 4 minutes.

From the obtained results and through the curves explanation, it’s clearly that a higher value in hardness was obtained as the dipping time and temperature of coating solution increased. The influence of phosphating time and immersion solution temperature are discussed in more details in Ref. It was found that increasing the solution temperature in the electrolyte results in accelerated coating growth.

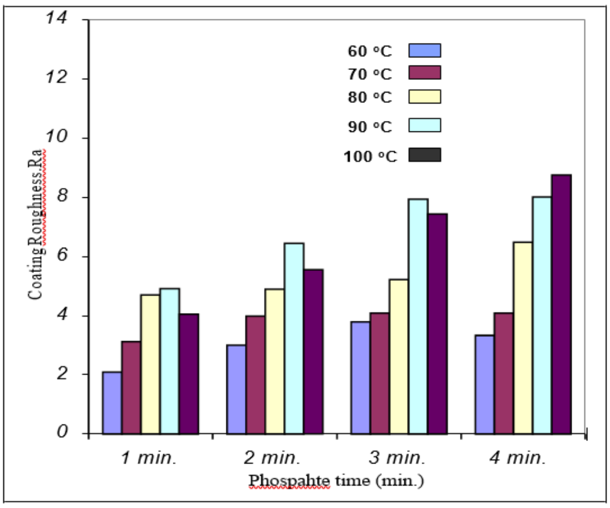

Figure 10. Coating roughness vs. phosphating time at different phosphate solution temperature

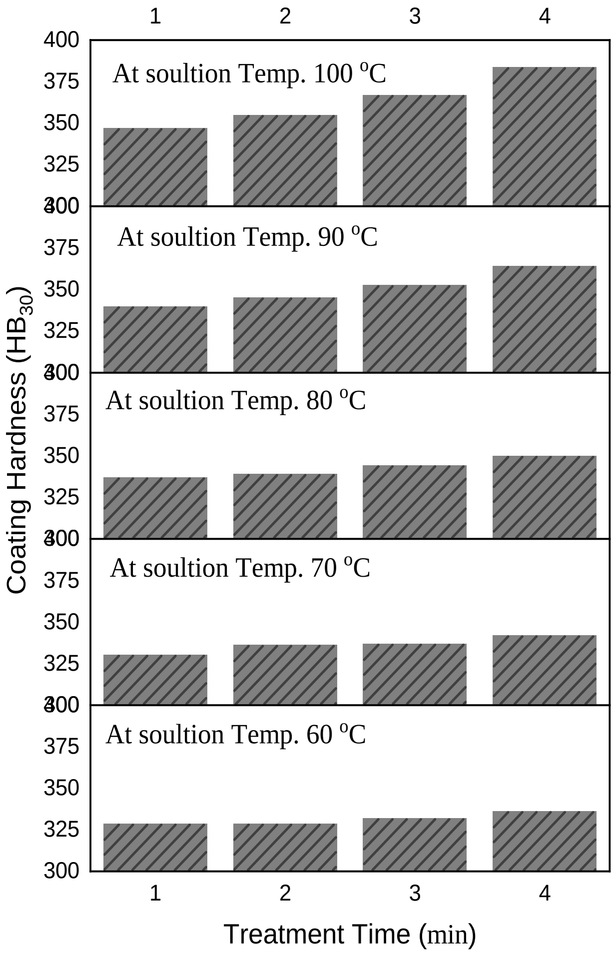

Figure 11. Coating roughness vs. phosphating time at different phosphate solution temperature

IV. CONCLUSIONS

By visual appearance, the colure of zinc phosphate coatings was lies between light grey and shine black. The results obtained of visual appearance tests, showed that the best finish can be found in conditions at phosphate solution temperature 90^{o}C for phosphate time 3 and 4 min respectively. Also, the results indicated that for all phosphating solution temperatures used in the process, the phosphate conversion layer thicknesses increase with increasing in treatment time. However, in the last time period, the coatings growth rate was reduced due to the dissolution and recrystallisation of the coating. The surface roughness and substrate hardness of phosphate coat layer increase as the phosphating time increase in all conditions of process temperatures.

This research studied the effect of phosphate time and solution temperature on the zinc phosphate layer. Therefore, the future scope of the current analysis is that to study the effect of primer (underpaint), by using top coat, and study the difference in performance properties of the top coat on the alloy steel surface with and without zinc phosphate coating.

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