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AN INVESTIGATION ON FATIGUE AND FATIGUE CORROSION BEHAVIOR OF Cr-Si SPRING STEEL AFTER Zn-Ni ELECTROPLATING USING WEIBULL STATISTICAL MODEL

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Abstract: *Compression springs were prepared from Cr-Si high strength spring steel and coated with pure Zn and Zn-Ni by electroplating process. The effect of baking after electroplating as well as applying an electroless nickel interlayer on the fatigue and fatigue corrosion of the springs was investigated. The results were analyzed using weibull statistical model. A considerable improvement (8%) in fatigue life of the electroplated springs with Zn-Ni was observed in the presence of Ni interlayer. In addition, baking of these electroplated springs improved fatigue life by 4%. The fatigue life under salt spraying conditions, however, has demonstrated remarkable reduction by 40%, 34% and 30% for Zn-Ni plating, backed and unbaked Zn-Ni plating containing Ni interlayer, respectively.*

Keywords: *Cr-Si spring steel, Zn-Ni alloy electroplating, Fatigue, Fatigue corrosion.*

1. INTRODUCTION

Springs, necessarily, tolerate severe fatigue conditions while being used under servicing circumstances. Therefore, production processes, from material selection to its assembly on the desired system, are leading to avoid reduction of spring fatigue life. Also, high tensile strength of spring steel will lead to increasing sensitivity to failure due to the dynamic loads [1-2].

Cr-Si high strength spring steel is widely used for fabrication of various spring types. High tensile strength is resulted in severe sensitivity to hydrogen embrittlement. Electroplating process causes hydrogen penetration due to the hydrogen evolution reaction (HER). When steel is placed into tensile situation under working conditions, this will cause sudden failure. In addition, electrochemical processes not only produce hydrogen atom but also reduce fatigue life under dynamic loading conditions due to the possibility of creating tensile residual stresses on the steel surface and high density of micro-cracks [3-4]. In conditions where there is a risk of hydrogen embrittlement, one of the suitable ways is de-embrittlement through baking treatment [5]. Based upon BS 1706-1990 and ASTM B850-98 standards, steel parts are heated in 190-220 °C, the duration of which depends on tensile strength of steel substrate and for Cr-Si spring steel is considered as 24 hours.

On the other hand, Zn-Ni alloy electroplating has been widely used for protection of steel parts against corrosion. Mostly, Zn-Ni alloy containing 10-12%Ni is able to reveal significant improvement in the control of corrosion rate in alloy deposits [6-14]. Many reports have demonstrated that applying an interlayer of electroless nickel would improve fatigue life in electroplated steels [3-4, 15-17]. However, improvement of substrate steel fatigue life by electroless nickel interlayer is only possible when this layer contains phosphorus amount less than 4% or between 10 to 12% because the interlayer will create compressive residual stress within the aforesaid chemical composition range that may lead to the increase in fatigue strength of steel substrate. Whereas that, 4 to 9% phosphorus range, nickel interlayer will create tensile residual stresses and will have undesired effects on fatigue life [17].

It is believed that one of the best ways to increase fatigue life in spring steel is shot peening of the spring surface before final coating. Shot peening process slows down the propagation of surface cracks as the result of dynamic tensile

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loads into the substrate through making a compressive residual stress in the surface. As such, shot peening through delaying in cracks formation and preventing from its development into the substrate structure will improve spring fatigue life [18]. Therefore, Cr-Si high tensile strength spring steel due to the sensitivity to surface crack and hydrogen embrittlement must be necessarily put into shot peening process. So, it seems that fatigue of Cr-Si spring steel is completely influenced by coating conditions and cracking due to hydrogen embrittlement.

In this paper, fatigue behavior of a compressive spring made of Cr-Si high strength spring steel electroplated by Zn-Ni was evaluated by using weibull statistical model. The main reason is the probability of results dispersal due to the fatigue test that can be attributed to the mechanism of diffusive hydrogen atoms into the metal structure as well as the variation of microstructural defects of substrate steel. Hillier & Robinson have successfully applied this model to describe Zn-Co alloy coating performance [19]. In this model, probability of non-failure of the samples is defined as follows:

$$P_s=1-P_f=e^{-x(N-Ni)} \qquad (1)$$

In which P_s is the non-failure probability of the sample, P_f is the failure probability, N is the number of cycles up to failure, and X is the shape parameter that is related to weibull curve slope. In addition, N_i is the minimum number of cycles that the sample tolerance to break. In other words, Ni is the number cycles in which failure probability of sample is equal to 1 and therefore $N=N_i$.

2. EXPERIMENTAL PROCEDURE

2. 1. Spring Samples

Figure 1 displays a spring that has been studied in this article. The samples were prepared from Cr-Si spring steel 55SiCr6, with 1920 MPa tensile strength, and 56RC hardness. Chemical composition of this spring is given in table 1. In addition, spring geometrical specifications are provided in table 2. The spring samples were exposed to shot peening process before electroplating in order to increase their fatigue strength.

Fig. 1. Studied spring in this research

Table 1. Chemical composition (%) of Cr-Si spring steel 55SiCr6

C	Mn	Cr	Si	P	S
0.55	0.68	0.64	1.44	0.009	0.007

Table2. Geometrical specification of studied spring

wire diameter	free length	internal diameter	external diameter	No. of coils
4.52mm	52.4mm	24.1mm	33.6mm	6.1

2. 2. Zn-Ni Electroplating

Alkaline bath with the chemical composition as shown in table 3 was used in this research for Zn-Ni alloy electroplating. In the bath, the temperature was controlled at 25°C, pH at 13 and current density at 3A/dm^2 for 10 µm deposit thickness of Zn-12%Ni. Experimental attempts revealed that suitable duration to achieve the mentioned thickness was 30 minutes. Some of the samples were gone through baking treatment in 200°C for 24 hours after alloy electroplating. According to ASTM B850 standard, baking treatment was carried out with time delay of less than 3 hours after electroplating.

Table 3. Chemical composition and characteristics of the Zn-Ni electroplating bath

characteristics	Composition of electrolyte (g/lit)					pH	temperature (°C)	current density (A/dm^2)
	$ZnCl_2$	$NiCl_2$	NaOH	Na_2CO_3	brightener			
amount	10	1	130	60	4	13	25	3

2. 3. Pure Zn Electroplating

A standard cyanide bath with the chemical composition and specifications listed in table 4 was used for pure Zn electroplating. In order to accomplish a coating with the thickness of 10 μm, electroplating duration was considered as 20 minutes after several attempts.

2. 4. Electroless Nickel Interlayer

To prepare a thin layer of electroless nickel with phosphorus containing less than 3% as an interlayer, an electroless alkaline bath was used with the chemical composition and specifications indicated in table 5. The aforesaid interlayer was deposited directly on the steel substrate before Zn-Ni electroplating. After several attempts, a deposit with 5μm thickness was gained within 30 minutes. It is necessary to mention that samples are washed in 10% nitric acid before nickel electroless process for activating the surface and make it ready to adsorb nickel deposits due to the improvement of wettability.

2. 5. Fatigue and Fatigue Corrosion Tests

Fatigue and fatigue corrosion tests on the springs were done within 31-42.5 mm oscillation amplitude with 25 Hz frequency range. As the free spring length is 52.4 mm, it is evident that aforesaid tests have been done within compressive stresses range. Finally, number of cycles up to failure in each test was determined and recorded. Totally 10 springs were tested in each of the studied conditions. To summarize, samples preparation procedure as well as the research steps are displayed in figure 2. Fatigue and fatigue corrosion tests were done by means of the existing devices in Iran FanarLool Company.

Fatigue corrosion test was done with the aim to examine the influence of environmental conditions on the springs' fatigue behavior made of Cr-Si Steel. For this purpose, a corrosive environment was applied consisting of 3.5% NaCl solution through spraying mechanism. Table 6 provides a summary of electroplating terms for the springs under fatigue and fatigue corrosion tests.

Table 4. Chemical composition and characteristics of the pure Zn electroplating bath

characteristics	Composition of electrolyte (g/lit)					pH	temperature (°C)	current density (A/dm^2)
	NaCN	ZnCN	NaOH	Na_2CO_3	brightener			
amount	47	61	79	15	4	12.3	25	3

Table 5. Chemical composition and characteristics of the Ni electroless plating bath

characteristics	Ni electroless solution composition (g/lit)					pH	temperature (°C)
	$NiCl_2.7H_2O$	NaH_2PO_2	NH_4Cl	NH_4OH	$Na_3C_6H_5O_7.5H_2O$		
amount	30	20	50	9	80	9.5	80

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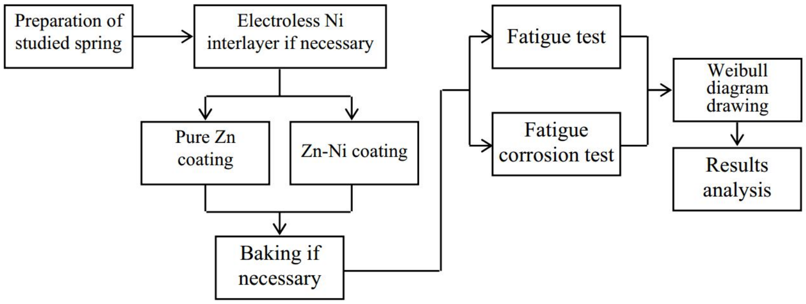

Fig. 2. Flowchart of samples preparation and research stages

Table 6. Final conditions of springs for fatigue and fatigue corrosion test

samples conditions	fatigue	fatigue corrosion
Zn-Ni	√	√
Ni/ Zn-Ni	√	√
Ni/ Zn-Ni/ baking	√	√
pure Zn	√	--
bare	√	--

3. RESULTS

3. 1. Results of Fatigue Test

Table 7 indicates the results of fatigue test and average number of cycles up to failure of different samples. Below, one would find more details on weibull diagram, for example for the samples without coatings. Results of fatigue test related to these bare samples are given in table 8. As in the present study, 10 samples were prepared in each condition, different values for Ln(P_s) are

Table 7. Results of fatigue test for number of cycles up to spring failure. In each case, the number of cycles up to failure has sorted from low to high.

samples conditions	number of cycles up to failure ($\times 10^7$)										
	1	2	3	4	5	6	7	8	9	10	**mean**
Zn-Ni	1.92	2.01	2.06	2.11	2.15	2.17	2.18	2.25	2.29	2.34	**2.15**
Ni/ Zn-Ni	2.19	2.24	2.25	2.26	2.29	2.33	2.38	2.39	2.41	2.44	**2.32**
Ni/ Zn-Ni/ baking	2.19	2.20	2.37	2.39	2.42	2.43	2.48	2.51	2.59	2.61	**2.42**
pure Zn	1.89	1.96	1.99	2.01	2.08	2.09	2.12	2.16	2.18	2.22	**2.07**
bare	2.70	2.78	2.88	2.89	2.97	2.99	3.01	3.06	3.09	3.10	**2.95**

Table 8. Results of fatigue test for bare samples based on the calculations of weibull model.

number of cycles up to failure	P_s	Ln(P_s)	x (shape parameter)
2.70×10^7	1	0	∞
2.78×10^7	0.9	-0.11	1.32×10^{-7}
2.88×10^7	0.8	-0.22	1.24×10^{-7}
2.89×10^7	0.7	-0.36	1.88×10^{-7}
2.97×10^7	0.6	-0.51	1.89×10^{-7}
2.99×10^7	0.5	-0.69	2.39×10^{-7}
3.01×10^7	0.4	-0.92	2.96×10^{-7}
3.06×10^7	0.3	-1.2	3.34×10^{-7}
3.09×10^7	0.2	-1.61	4.12×10^{-7}
3.10×10^7	0.1	-2.3	5.76×10^{-7}

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gained as evidenced in table 8. Applying equation (1), it is showed the probability of springs' non-failure in fatigue test for the bare samples as $Ln(P_s)=-X(N-2.7\times10^7)$. For example, probability of non-failure of the samples fatigue up to 2.7×10^7 cycles is 100% since no sample has failed under lower cycle. Another example: Non-failure probability up to 3.06×10^7 cycles is 30% because 70% of the samples have failed before this value.

3. 2. Influence of Zn-Ni Electroplating

Figure 3 displays weibull diagram for the number of cycles up to failure for the samples having Zn-Ni and pure Zn coatings in comparison with the bare springs. Average number of cycles for coated springs with Zn-Ni in comparison with pure Zn is around 4% more, while both of these coatings have indicated less number of cycles than the bare springs. These results indicate that electroplating of springs in both conditions affect fatigue performance badly with no significant difference.

3. 3. Influence of Electroless Nickel Interlayer

Figure 4 displays weibull diagram of fatigue test results to studying the influence of electroless nickel interlayer. Presence of this interlayer before Zn-Ni has increased the number of cycles

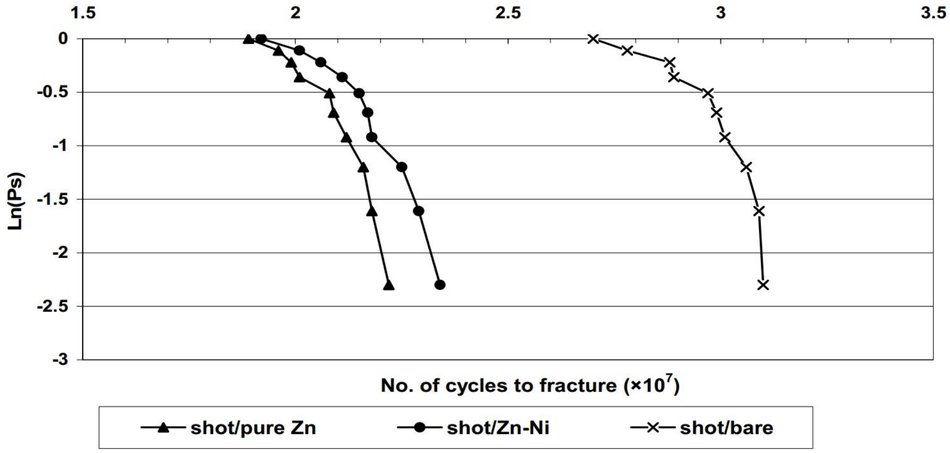

Fig. 3. Weibull diagram for number of cycles up to failure in fatigue test for springs with pure Zn and Zn-Ni coating

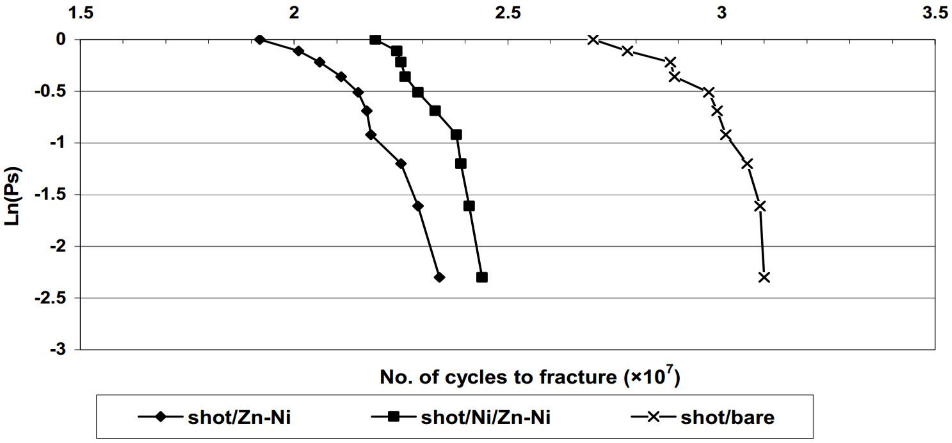

Fig. 4. Weibull diagram for number of cycles up to failure in fatigue test for assessment of electroless Ni interlayer effect

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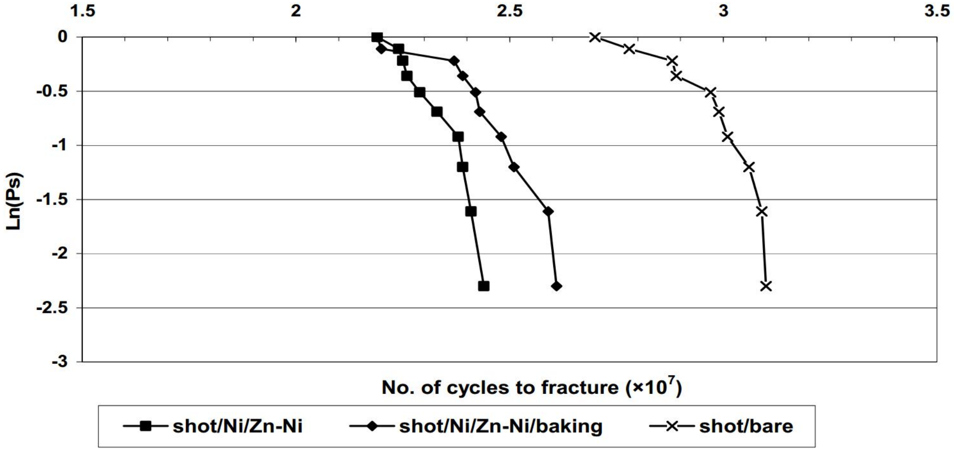

Fig. 5. Weibull diagram for number of cycles up to failure in fatigue test for assessment of baking treatment effect

by 8% to the springs' failure. In other words, presence of interlayer has more influence than Zn alloying.

3. 4. Influence of Baking Treatment After Electroplating

Figure 5 indicates results of the influence by baking treatment after electroplating in the springs consisting of nickel interlayer. By average, baking the electroplated springs with Zn-Ni containing nickel interlayer, leads to 4% increase in the number of fatigue cycles up to the springs failure.

3.5. Influence of Environmental Conditions

Table 9 provides the results of fatigue corrosion test on the samples. These data have been used to draw weibull statistical diagrams. For a better comparison, fatigue corrosion test results are shown in figure 6 beside fatigue test results. It is evident that in the presence of a corrosive environment, the fatigue has remarkably reduced number of cycles up to failure. However, average reduction of the above for the springs with Zn-Ni plating is 40%, the same for those springs with Zn-Ni plating and nickel interlayer is 30%, and this for Zn-Ni samples including nickel interlayer and baked is 34%.

4. DISCUSSION

4. 1. Influence of Zn Alloying with Nickel

As it can be seen in figure 3, average number of cycles up to failure of those springs plated with Zn-Ni is 4% more than pure Zn plating, although both pure Zn and Zn-Ni coatings indicate less number of cycles up to failure in comparison with the springs with no plating. This little improvement of fatigue behavior as the result of

Table 9. Results of fatigue corrosion test for number of cycles up to spring failure. In each case, the number of cycles up to failure has sorted from low to high.

samples conditions	number of cycles up to failure ($\times10^7$)										
	1	2	3	4	5	6	7	8	9	10	**mean**
Zn-Ni	1.19	1.25	1.26	1.28	1.29	1.30	1.33	1.33	1.38	1.40	**1.30**
Ni/ Zn-Ni	1.53	1.55	1.59	1.61	1.62	1.62	1.68	1.69	1.71	1.75	**1.63**
Ni/ Zn-Ni/ baking	1.49	1.54	1.56	1.57	1.59	1.61	1.62	1.63	1.69	1.72	**1.60**

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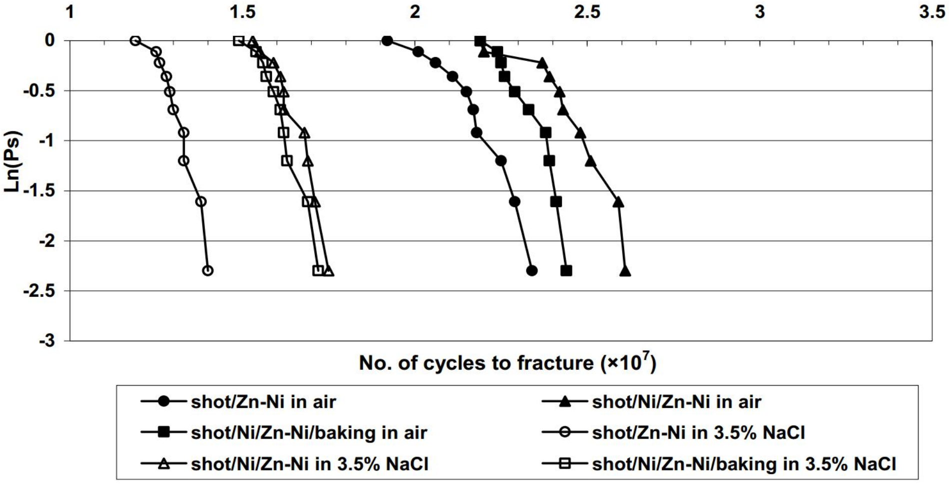

Fig. 6. Weibull diagram for number of cycles up to failure in fatigue corrosion test for assessment of environmental conditions effect

alloying Zn coating with nickel indicates that springs fatigue failure, mostly the coated type, is affected by the type of electroplating process. In fact, electroplating leads to the downfall of springs fatigue performance with almost similar conditions. Very little improvement due to the presence of nickel in Zn coating structure is considered to be the result of formation of a nickel enriched layer in interface coating/substrate. Existence of this layer has been approved through numerous reports [5, 19-20]. This thin layer acts as a physical obstacle against hydrogen diffusion as the result of electroplating process. As Zn-Ni alloy deposition mechanism is anomalous, obviously this Ni enriched layer has been created at the early stages of electroplating through a normal process. This small difference in the performance of pure Zn and Zn-Ni coatings indicate that fatigue performance in these springs highly depends upon shot peening before electroplating. Shot peening process creates a thin layer including compressive residual stresses on the substrate surface, by which penetrative hydrogen decreases due to the reduction of lattice parameter and shrinkage of the empty accessible space on the one hand and creates remarkable time delay in hydrogen diffusion on the other hand. One of the reports confirms that shot peening is able to create time delay up to 400% [18]. In addition, when the crack tip reaches the region of compressive residual stresses, cracks propagation rate slows down remarkably. Therefore, shot peening will lead to the reduction of microcracks propagation rate as the result of tensional stresses under servicing conditions. Figure 7 displays a schematic view of crack propagation in the peened steel substrate with a multilayer coating system.

4. 2. Influence of Electroless Nickel Interlayer

Figure 4 shows the influence of electroless nickel interlayer on the springs fatigue life. As it was mentioned, presence of nickel interlayer before final Zn-Ni coating increases the number of cycles up to failure by around 8%. In other words, presence of Ni interlayer has more effect than Zn alloying with nickel. This also confirms the positive influence of Ni enriched layer formation in the initial stages of electroplating through a normal mechanism. The influence of Ni interlayer on the improvement of fatigue life is justified as follows.

Nickel interlayer created by electroless alkaline bath involves compressive residual stresses [21]. While Zn-Ni deposited by electroplating bath, involves tensile residual stresses [22]. Hence, although the existence of a Ni enriched layer is able to resist against hydrogen diffusion as a physical obstacle, but due to an imbalance between compressive

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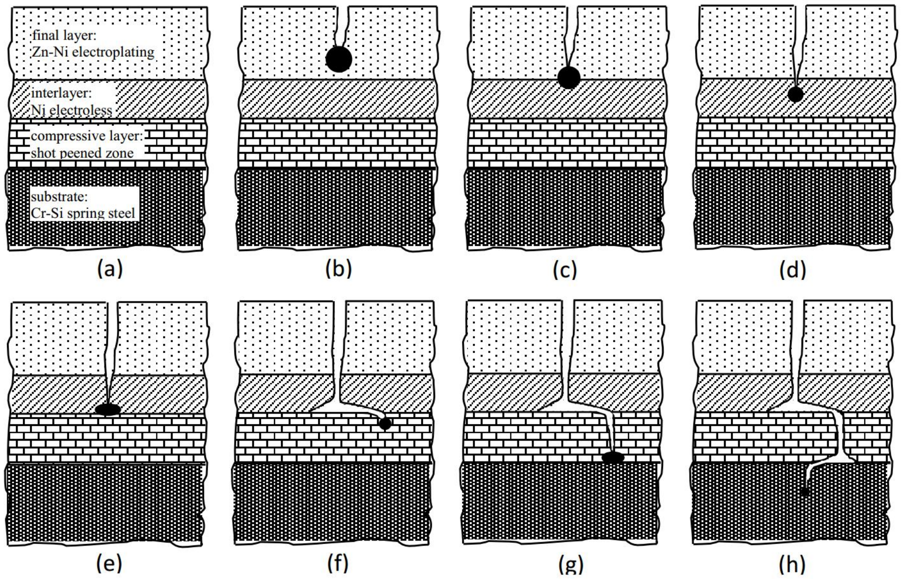

Fig. 7. Schematic of crack propagation in steel substrate in presence of a multilayer coating system containing Zn-Ni alloy and interlayer nickel. (This figure was developed based on research-work of Nascimento [15])

residual stresses in the interlayer and tensile residual stresses in the alloy deposits, the interface between interlayer and final coating will face unstable situation and finally its high energy level will provide a suitable driving force to move hydrogen atoms from the upper layer towards interface. Therefore, although the hydrogen diffusivity is low in nickel interlayer, but the amount of hydrogen penetrates into the substrate. Therefore, Ni interlayer cannot prevent completely from hydrogen diffusion and remove the risk of hydrogen embrittlement. According to figure 4, presence of Ni interlayer somewhat increases the number of fatigue cycles up to failure but there is still a large distance to the bare spring conditions.

4. 3. Influence of Baking Treatment After Electroplating

It is clear in figure 5 that baking treatment in those samples having Zn-Ni plating and Ni interlayer has improved springs fatigue life by around 4%. Altogether, baking treatment with the presence of Ni layer has improved fatigue performance by 12% more than Zn alloying with Ni.

Taking into account that baking treatment is done in 200°C and no structural and physical structure changes are made in this temperature range [23-25], the influence of baking treatment is attributed to a suitable thermodynamic condition for the desorption of diffusive hydrogen. Numerous reports exist in which the existence of dense microcrack network in interface between Zn-Ni alloy coating and steel substrate is approved [12-13, 22, 26]. Existence of these cracks is due to the residual stresses in non-epitaxial interface of coating / substrate. As the result of baking treatment, severe difference between thermal expansion coefficient of Zn-Ni alloy and Cr-Si steel will intensify the above mentioned cracking network. Consequently, hydrogen diffusion is expedited through this dense network that will lead to the more desorption of hydrogen atoms and increase in the number of cycles up to failure in springs fatigue test. However, the influence of baking treatment is not outstanding in the presence of Ni interlayer; since crack tip reaches to the interface Zn-Ni/Ni electroless after the development of surface cracks throughout Zn-Ni alloy coating thickness. Since hardness of the Ni interlayer

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having residual compressive stresses is more than Zn-Ni layer, entrance of crack tip plastic zone to the interlayer will face with difficulty (Figure 7.c and 7.d). This may reduce and even stop the crack propagation rate in the interface Zn-Ni/Ni. As such, dense microcracks network will not develop from interlayer to the steel substrate due to the prevention by nickel interlayer. Whereas this integrated network provides a suitable direction for the exit of penetrative hydrogen towards free surface. Therefore, access level of penetrated hydrogen atoms into Cr-Si steel to the free surface decreases and finally a small amount of hydrogen will remain in steel substrate structure. As the result, Zn-Ni samples with Ni interlayer will indicate only 4% improvement in fatigue performance after baking treatment.

4. 4. Influence of Environmental Conditions on Springs Fatigue Performance

The results of fatigue corrosion test are displayed in figure 6 by weibull diagram. It is clear that springs' fatigue performance with the presence of a salty corrosive environment has failed significantly. However, average value of this reduction is different for various conditions. Amount of the aforesaid reduction for those springs with Zn-Ni coating is around 40%, the same for Zn-Ni with Ni interlayer is around 30%, and 34% approximation is recorded for the springs plated with Zn-Ni and Ni interlayer and baked. These differences are evident under circumstances in which loading frequency is similar at all cases. It means the available time for corrosive particles attracted to the crack tip to occur electrochemical reactions is similar. As the surface absorption of environmental particles may lead to the electrochemical corrosion reactions at the crack tip under fatigue corrosion conditions, increase in the crack propagation rate and consequently the reduction of fatigue life are expectable. In addition, the high strength of Cr-Si steel and its severe sensitivity to hydrogen embrittlement will increase the fatigue cracking propagation rate. In fact, there is a synergic effect between fatigue dynamic stresses and electrochemical reactions at the crack tip that will form crack growth mechanisms together [27].

The maximum of reduction in the number of cycles up to the spring failure under fatigue corrosion conditions with 40% belongs to the springs only plated with Zn-Ni. This subject is justified as follows; Zn-Ni alloy coating surface consists of a network of microcracks that are integrated. On the other hand, corrosion is considered to be a predominant factor under fatigue corrosion conditions, especially in chloride environments [27]. Suitable path for the cracking propagation in those samples only plated with Zn-Ni have preexisted such that corrosion reactions are suitably able to cause faster fatigue cracking. In other words, crack initiation stage is omitted due to the existence of earlier microcracks.

However, the minimum of fatigue performance downfall under corrosive circumstances with the value of 30% belongs to the springs with Zn-Ni coating and presence of Ni interlayer. Hardness of this interlayer is more than the final Zn-Ni layer. Therefore, surface microcracks network stops as it reaches the above region. As such, since the active paths for crack propagation face an obstacle called nickel interlayer, it will face the reduction of suitable regions for corrosive particles attraction at the crack tip. This will reduce corrosion reaction rate at the crack tip and increase fatigue life more than those samples with no Ni interlayer.

Reduction of the number of cycles up to the spring's failure plated with Zn-Ni including Ni interlayer and baked is approximately 34% under corrosive environments in comparison with air. It seems that baking treatment in the springs plated with Zn-Ni and Ni interlayer had a reverse influence. This is just because an amount of hydrogen penetrated into the substrate microstructure exits through suitable paths in the surface microcracks of Zn-Ni coating as the result of baking treatment. With the presence of hard Ni interlayer, dense microcracks network will not propagate inside the interlayer and reduction in the access of diffusive hydrogen atoms into the substrate will increase the amount of remaining hydrogen in steel substrate in comparison with those springs lack of any interlayer. Existence of the above remaining hydrogen may lead to the empowerment of

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fatigue corrosion cracking through hydrogen embrittlement mechanisms. As such, it is expected that fatigue life reduction in the springs plated with Zn-Ni including nickel interlayer and baked under corrosive circumstances is more than unbaked springs but less than those springs only plated with Zn-Ni.

5. CONCLUSIONS

1. Number of cycles up to the failure point of Cr-Si compressive springs electroplated with Zn-12%Ni has indicated 4% of improvement more than the similar samples plated with pure Zn in fatigue test.
2. Fatigue life in Zn-Ni plated springs, in the presence of electroless nickel interlayer, indicates 8% increase. In other words, the influence of Ni interlayer is more than Zn alloying with Ni.
3. Baking of electroplated springs with Zn-Ni alloy in the presence of nickel interlayer increases the number of cycles up to the spring failure in fatigue test by 4%.
4. Number of cycles up to the spring failure under salt spraying conditions in fatigue corrosion test has indicated a remarkable reduction in comparison with fatigue test in air. In such case, average amount of the above reduction for the springs only plated with Zn-Ni is around 40%, the same for Zn-Ni plating and Nickel interlayer is around 30%, and this figure for Zn-Ni plating including nickel interlayer and baked is around 34%.

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