1. Introduction

Hot dip zinc coatings are extensively used for the protection of carbon steel wires. In such cases, the more active zinc metal corrodes preferentially than the steel wire by a cathodic reaction that prevents steel from undergoing anodic corrosion reaction. In a conventional hot dip galvanizing process, a steel wire is chemically cleaned, fluxed, and then immersed in a molten zinc bath at a temperature of about 450 °C [1]. Before the immersion in the molten zinc bath, the steel wire is fluxed in an aqueous solution containing chloride salts to avoid any surface oxidation and contamination. The thickness of hot dip galvanized coatings depends on withdrawal speed and the silicon concentration at 450 °C, which is related to the total iron loss of the substrate. If the withdrawal speed is too slow, a uniform unalloyed zinc layer is formed, while in the case of faster withdrawal speed, an uneven coating is formed. At the exit from the molten zinc bath, the wiping systems accurately control the thickness of coating on the surface by removing the excess of molten zinc. Then the steel wire is spray quenched with a mixture of air and water to obtain a bright shiny finish.

Hot dip galvanized coating formation can be described as a diffusion process. Zinc diffuses into the steel and iron diffuses into the zinc. As the diffusion coefficient of zinc is higher than that of steel, it is believed that zinc readily diffuses into the steel and forms intermetallic compounds. A hot dip galvanized coating consists of a heterogeneous assembly of different phases which are formed due to metallurgical reactions between iron (Fe) and zinc (Zn) when a steel wire is immersed into molten zinc. After solidification, the coating consists of an outer layer of 100% zinc (η-eta layer) and inner layers called alloy layers consisting of intermetallic phases of iron and zinc such as zeta (ζ) layer (94% Zn–6% Fe), delta (δ) layer (90% Zn–10% Fe), and gamma (Γ) layer (75% Zn–25% Fe). These intermetallic layers are relatively harder than the underlying steel and provide exceptional protection against coating damage [1].

The extent of effective life of hot dip zinc coating not only depends on the coating composition but also on the metallurgical characteristics of the coating. Among the types of failure encountered in galvanized steel spring wires, fatigue is the most common one. There are many factors that determine the performance of zinc coated steel springs. Steel chemical composition, microstructural properties, steel surface quality, coating thickness, coating quality, and coating microstructure are among these [2–4]. In particular, surface defects and coating defects can create notch effects in spring wire production. The springs running under repeated stresses are broken by the development of the cracks that develop with time and cause damage [5–7]. This study is a detailed failure analysis of galvanized high carbon (0.83%) steel spring wires, which developed coating cracks during the fatigue test performed as a quality control at the end of the manufacturing process.
2. Experimental procedure

Chemical composition of steel wire is presented in Table I. After austenitising, the steel wire is subsequently cooled in a patenting heat treatment process where the austenitised wire is cooled in a bath of molten lead and held at a temperature below 500°C to form fine pearlite which is considered to be the suitable microstructure to assure good cold-formability. Heavily deformed wires exhibit a plastic strain localisation and the as-draw wire contains residual stresses, which are sufficient to produce a hardening effect during a post drawing tensile test. Changes in ductility can occur during the deformation process in wire drawing and forming. After a series of surface treatment steps, the wire rods (0.83C, ø: 9–10 mm; BS EN ISO 16120-1.2011 standards) are coated in suitable thicknesses in the molten zinc bath (at 450°C). Production process steps are as follows: pay-off → acid pickling → hot water cleaning → fluxing → drying → hot-dip galvanizing → wiping system → take-up. The zinc coated (hot dipping) (Fig. 1) wires are brought to the appropriate diameter range and then brought into the form of a spring.

![Diagram of continuous galvanising process for wires](image)

Fig. 1. Continuous galvanizing process for wires (schematic) [6].

| Element | C  | Mn | Si | P   | S   |
|---------|----|----|----|-----|-----|
| wt%     | 0.84| 0.59| 0.22| 0.014| 0.017|
| Element | Cu | Cr | N  | Ni  | V   |
| wt%     | 0.015| 0.034| 0.005| 0.016| 0.001|

Metallographic analyses were carried out on longitudinal and transversal cross-sections cut from the wires in different positions. Samples were grinded, polished, and etched with 3% nital (HNO₃) to reveal the microstructure of both coating and steel. The microstructures were examined using a optical microscope (OM) and scanning electron microscope (SEM). Fatigue test unit adjusts machine test stroke (100–300 mm) and length of the spring set position, the tension spring or compression spring fixed in the fixture (30–500 kN), set test times and speed (25 dev/dak.), proceed with test until the broken spring bounce fixture. Fatigue tests applied to the springs are required to withstand \( \approx 2 \times 10^6 \) cycles. After the fatigue test, failure analysis was performed on broken springs (\( \approx 10^3 \) cycles). Fracture surfaces and zinc coatings on steel springs were examined in detail under a microscope.

3. Results and discussion

Main causes of breakage of fatigue are: improper metallurgy, chemical composition, incorrect forming profile, excessive wire reduction, segregation, decarburisation, porosity, and other surface defects. It has been observed that the springs do not have sufficient fatigue life. Cracks and deformation traces are observed on steel spring surfaces (Fig. 2). In this context, input wire rod quality control tests were started to determine the microstructural discontinuities that will affect the spring failure.

![Surface of the damaged spring wire](image)

Fig. 2. Surface of the damaged spring wire.

Decarburisation and segregation can significantly reduce strength and fatigue life of the steel. Decarburisation problems must be avoided. It is necessary to control decarburising depth for both rod and wire. Microstructural discontinuity was not observed as a result of the tests made in accordance with the relevant standards (decarburisation depth: ISO 3887:2003 (ISO 3887:2017); segregation: BS EN ISO 16120-1:2011; inclusion: ISO 4937:2013; surface discontinuity: BS EN ISO 16120-1:2011) in Fig. 3.
It is understood that typical fatigue fracture damage occurs when the fracture zone is examined, as shown in Fig. 4. It was determined that the crack initiation occurred in regions near the surface. It is observed that the discontinuities on the surface cause crack formation and that the fracture occurs after increasing repeated stresses. It is predicted that these surface defects will occur during wire drawing operations.

Coating thickness is between 30–35 µm. Various intermetallic (Fe–Zn) phases were detected in the coating structure. Vertical cracks are detected along the transverse section of the examinations made on the coating surface. There are also metallurgical phase differences along the coating thickness. This indicates that the cooling process is not suitable. Adhesion properties at the interface between the coating and the substrate are very poor, as shown in Fig. 5. As a result of EDX analysis,
sis in the coating section, it was assigned to Cl and O elements. This shows that the surface preparation before the coating process is not sufficient, as reported in Fig. 6. Corrosive products (FeCl, FeO based) occurred at the interface as a result of acidic reactions.

Fig. 6. EDX line analysis of the zinc coating layer on steel wire.

4. General conclusions

Spring wire materials typically contain 0.45%–0.85% C and 0.60% Mn. The steel wire undergoes a patenting (high-temperature process conducted at 450°C–570°C) process then it is cold-drawn which increases strength without loss of ductility. Findings from the failure analysis study on this high carbon steel spring wire are shown below. Surface and subsurface metallurgical defects act as stress risers (concentrators) and the surface is subjected to the highest tension, bending and torsion stresses [7]. Tensile stresses developed during the cooling after galvanizing. Generally, a fatigue crack will start at a surface defect on the spring. It is a fatigue fracture cracking that causes this workpiece to suffer failure due to repeated tensile loads. When the fracture surface is examined, it clearly shows signs of fatigue. These cracks originate from surface defects and develop afterwards, leading to breakage.

The importance of surface preparation has been emphasized once again in this study. Hot dip galvanizing is a most common coating application of protection against corrosion for wire springs. Due to the residence time of the steel in the melt zinc bath, Fe and Zn elements form an intermetallic diffusion layer. In the intermetallic layer, the Zn and Fe concentrations change and the hard and brittle phases depending on the cooling rate can occur. The brittle phases cannot withstand stresses against fatigue loads and as a result, cause high stresses on the surface, which lead to microcracks. The influence of silicon is remarkable after longer dipping times and slow withdraw speeds. The silicon equivalent in the steel composition must be within a certain range and should be kept under control. Hot dip operations and surface quality prior to forming affect the corrosion and fatigue performance of the spring wire. The adhesion properties of the zinc coating are impaired after unsuitable surface treatments. During the drawing process, intense deformation and cracks occur in the coating layer. This situation triggers crack formation on the wire surface and decreases the fatigue life.

As a result of diffusional effects and process conditions, the stresses and undesired phases on the interface of the zinc/steel cause notching effect on the spring and shortening of the fatigue life.

References

[1] S.M.A. Shibli, B.N. Meena, R. Remya, Surf. Coat. Technol. 262, 210 (2015).
[2] H.J. Gasterich, K.E. Hagedorn, R. Kaspar, O. Pawelski, Steel Res. 12, 125 (1993).
[3] S. Beretta, M. Boniardi, Int. J. Fatigue 21, 329 (1999).
[4] A.V. Olver, D. Wilson, P. Shaun, J. Crofton, Eng. Fail. Anal. 14, 1224 (2007).
[5] F. Berto, S.M.J. Razavi, M.R. Ayatollahi, F. Mutingham, Proced. Struct. Integrity 3, 77 (2017).
[6] M. Gelfi, L. Solazzi, S. Poli, Materials (Basel) 10, 264 (2017).
[7] L.C.F Canale, R. Penha, G. Totten, Int. J. Microstruct. Mater. Prop. 2, 262 (2007).