MARMARA UNIVERSITY
INSTITUTE FOR GRADUATE STUDIES
IN PURE AND APPLIED SCIENCES

ELECTROPLATED COATING OF CAST STEEL AND ZAMAK PARTS BY ACIDIC ZINC NICKEL

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MASTER THESIS
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ERKUT YILMAZ, a Master of Science student of Marmara University Institute for Graduate Studies in Pure and Applied Sciences, defended his thesis entitled "Electroplated coating of cast steel and zamak parts by acidic zinc nickel", on June 18, 2013 and has been found to be satisfactory by the jury members.

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I want to thank firstly my advisor Prof. Dr. Ayhan MERGEN who always helped and supported me. Then, I want to thank my family and my wife .

June, 2013 **Erkut YILMAZ**

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ÇELİK DÖKÜM VE ZAMAK PARÇALARIN ELEKTROLİZ YOLUYLA ASİTLİ ÇİNKO NİKEL KAPLANMASI

Yoğun korozif ortamlarda araçların fren sistemlerinde kullanılan fren kumpası gibi yüksek karbon içerikli döküm parçalar, düşük akım verimine sahip elektrolitik alkali çinko kaplamalar ile kaplanamamaktadır. Döküm parçalar yüksek akım verimli elektrolitik asitli çinko kaplamalar ile kaplanabilmektedir fakat asitli çinko kaplamaların korozyon dirençleri alkali çinko kaplamalardan daha düşüktür. Bundan dolayı tez çalışmamız döküm ve zamak parçalar üzerine hem akım verimi hem de korozyon dayanımı yüksek bir kaplama yapmayı amaçlamaktadır. Elektronegativite farkından dolayı; yapısında ağırlıkça % 12 - 16 arasında nikel içeren çinko nikel alaşım kaplamalar, yapısında % 100 oranında çinko içeren kaplamalara kıyasla 8 kat daha yüksek korozyon dayanımına sahiptir. Bu nedenle asitli çinko nikel alaşım kaplamalar yüksek akım veriminden dolayı parçanın her bölgesini iyi kaplayabilme yeteneğine ve yüksek korozyon dayanımına sahiptir.

Tez çalışmasında öncelikle ağırlıkça % 12 - 16 arasında nikel içerdiği XRF ile analiz edilen asitli ve alkali çinko nikel alaşım kaplamalarının farklı akım yoğunluklarında kaplanarak, faz, yüzey morfolojisi ve kristal yapıları SEM ve FIB resimleri ile incelenmiştir. Sonrasında 2,5 A/dm^2 'lik akım yoğunluğu ile alkali ve asitli çinko nikel alaşım kaplama ile kaplanmış döküm ve zamak parçalarının ASTM B-117 tuz testi öncesi, 240 ve 480 saat sonrası kaplama yapısındaki değişimler optik mikroskop ve SEM resimleri ile incelenmiştir.

Sonuç olarak; incelemeler sonrası asitli ve alkali çinko nikel elektrolit banyolarında yapılan döküm ve zamak parçalar üzerine kaplanan çinko nikel alaşım kaplamaların yüzey morfolojisi, gamma faz yapıları ve korozyon dayanımları birbirine benzemektedir.

June, 2013 **Erkut YILMAZ**

ABSTRACT

ELECTROPLATED COATING OF CAST STEEL AND ZAMAK PARTS BY ACIDIC ZINC NICKEL

The casting parts which have high carbon content as the composition and are used in high corrosive atmosphere such as brake caliper are not coated by alkaline coating with low current efficiency. They can only be coated by acidic zinc coating with high current efficiency but its corrosion protection is lower than alkaline zinc coating. Therefore the requirement is both high current efficiency and high corrosion protection coating for castings. Due to the electronegativity differences, zinc nickel alloy deposits which contain nickel between 12 - 16 wt.% in the alloy have approx. 8 times higher corrosion resistance than pure zinc deposits. Because of the fact that acidic zinc nickel alloy coatings have good coverage on the complex shape parts due to the high current efficiency, good adhesion and also high corrosion resistance properties.

In the thesis, firstly acid and alkaline zinc nickel alloy deposits containing nickel between 12-16 wt. % in the alloy were analyzed by XRF and their surface morphology, crystal structure and phases contents were investigated by SEM, FIB and XRD. Then alkaline and acid zinc nickel alloy deposits which were coated with 2,5 A/dm^2 current density were investigated by optical microscope and SEM before salt spray test and after 240 and 480 h ASTM B-117 neutral salt spray test.

As a result of these investigations it can be stated, that the acidic and alkaline zinc nickel electrolytes generated zinc nickel alloy coatings on casting and zamak parts with desired properties in terms of surface morphology, texture and gamma phase formation. The examined deposits showed good performance in terms of nickel incorporation for a production relevant current density spectrum.

June, 2013 **Erkut YILMAZ**

SYMBOLS

g : Gram

Kg : Kilogram

I : Current in ampere (A)

R : Resistance in ohms (Ω)

Ml : Mililiter

t : Time (s)

T : Temperature (°C)

V : Voltage in volts (V)

ABBREVIATIONS

EDS **:** Energy Dispersive Spectroscopy

FIB **:** Focus Ion Beam

I **:** Current

R **:** Resistance

SEM **:** Scanning Electron Microscopy

V **:** Voltage

XRD **:** X-Ray Diffraction

XRF **:** X-Ray Fluorescence

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1. INTRODUCTION

1.1. The Aim of The Thesis

The casting and zamak parts are used commercially in the industry but some applications are exposed to highly corrosive atmospheres. One of these applications is brake calipers for casting that are used in brake system of nearly all cars. The other is windows frame parts made of zamak that is a family of alloys with a base metal of zinc and alloying elements of aluminum, magnesium, and copper. The casting parts have high carbon content and both them are used in high corrosive atmosphere and they should be coated with a protective layer to increase lifetime and to decrease any catastrophic failure. However these parts are not coated by alkaline coating with low current efficiency. Because they can be only coated by acidic zinc coating with high current efficiency although, its corrosion protection is lower than alkaline zinc coating. The requirement for coating of these casting parts is both high current efficiency and high corrosion protection coating for castings. Due to the electronegativity differences, zinc nickel alloy deposits which contain nickel between 12 - 16 wt.% in the alloy have approx. 8 times higher corrosion resistance than pure zinc deposits. Because of this fact, acidic zinc nickel alloy coatings have good coverage on the complex shaped casting parts due to the high current efficiency, good adhesion and also high corrosion resistance properties.

In this thesis, acidic and alkaline zinc nickel alloy deposits which contain nickel between 12 - 16 wt. % was analyzed by XRF, SEM and FIB. Then casting and zamak parts are coated with alkaline and acidic zinc nickel alloy deposits at 2,5 A/dm^2 current density and they were investigated by optical microscope and SEM and XRD before and after neutral salt spray test (240 and 480 h ASTM B-117)

In recent years, due to increasing application areas of functional zinc nickel coating with corrosion resistance, affinity was increased to these coatings. Therefore it causes to investigate and struggle to develop new zinc nickel alloy coatings. Electrolytic coating industry is so active and automotive, white goods, defense industry, wind turbine and aerospace industry trigger new research and development in recent years.

1.2. Background

1.2.1. Electroplating

Electro-deposition of an adherent metallic coating onto a substrate which will produce a product with properties and dimensions, which differ from that of the basis material. The base or substrate is usually made from a metal or a metal alloy, although selective non-metals may also be coated. The coating must be adherent as a fixed part of the article. The combination of basis material and coating must fulfill specified properties regarding function and usability in dependence of the required application.

1.2.1.1 Electroplating history

Historical development of electroplating is given in **Table 1.1**. Luigi Galvani, Italian anatomist and physicist, discovered that muscle and nerve cells "produce" electricity (1737-1798). He was the first investigator to appreciate the relationship between electricity and animation. Typically credited with the discovery of bioelectric. The phenomenon he discovered during an experiment with a dead frog was dubbed as "galvanism". Galvani´s name survived e.g. in the term "galvanization".

Alessandro Volta, Italian professor of physics, known especially for the development of the electric battery (1745 - 1827). In 1775 he devised the electrophorus, which produced a static electric charge. He studied the chemistry of gases and discovered the methane. In 1800 he developed the so called "voltaic pile", a forerunner of the electric battery (**Figure 1.1**). He determined that the most effective pair of dissimilar metals to produce electricity was zinc and silver. In 1881 the important electrical unit, the "Volt", was named in his honor.

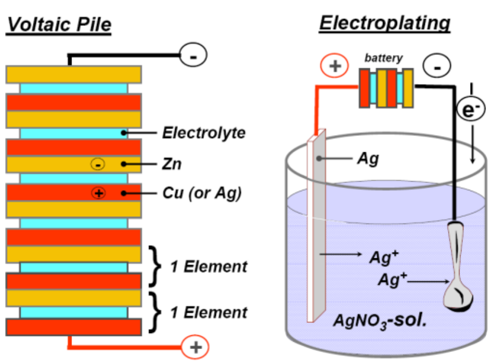

Figure 1.1. Voltaic pile and electroplating.

Michael Faraday, British physicist and chemist, contributed significantly to the fields of electromagnetism and electrochemistry (1791-1867). He is referred as the greatest experimentalist in the history of science. It was largely to Faraday´s efforts that electricity became viable for use in technology. He discovered the laws of electrolysis and popularized terminology as anode, cathode, electrode and ion. The SI unit of capacitance, the farad, is named after him, as is the Faraday constant, the charge a mole of electrons (about 96.485 coulombs).

Georg Ohm, German physicist, discovered Ohm´s Law (1789-1854). It states, that the potential difference between two points along a connected path and the current flowing through it are proportional at a given temperature.

$$I = \frac{V}{R} \text{ or } V = I \times R \text{ or } R = \frac{V}{I}$$

Table 1.1. Historical development of electroplating.

<1800 >1800 >1850		1917-1945	>1969	>1969
← Galvanic Cell →		Anodizing Conversion Coating Chromium Bronze Alloy Nickel Plating Chem. Milling Phosphating	Electrolytic Paint Electroless Nickel Electro Polishing Vacuum Metallizing Mechanical Plating Electrostatic Plating Plating on Plastics Powder Coating	Improvement of all coating Processes Ban of several metals and components (RoHS, WEEE) Development of more environment protecting products and processes
Polishing Grinding Painting Heat Treat Tempering	Silver Gold Copper Brass			

1.2.2. Electroplating vs Electroless Plating

Electroplating can be compared with electroless plating methods (**Figure 1.2**). Advantages and disadvantages of electroplating and electroless plating methods are compared in **Table 1.2**. Both of the methods have different advantages and disadvantages.

Table 1.2. Electroplating vs electroless plating

Properties of electroplating	**Properties of electroless plating**
Most common finishing process	More expensive
DC current plus metal ions	More uniform coating
Inexpensive	Plating on non-conductors
Easy	Control magnetism
Safe	Hard, wear resistant coatings
Conductive surfaces and non-conductors can be plated	Wire bonding in electronics industry

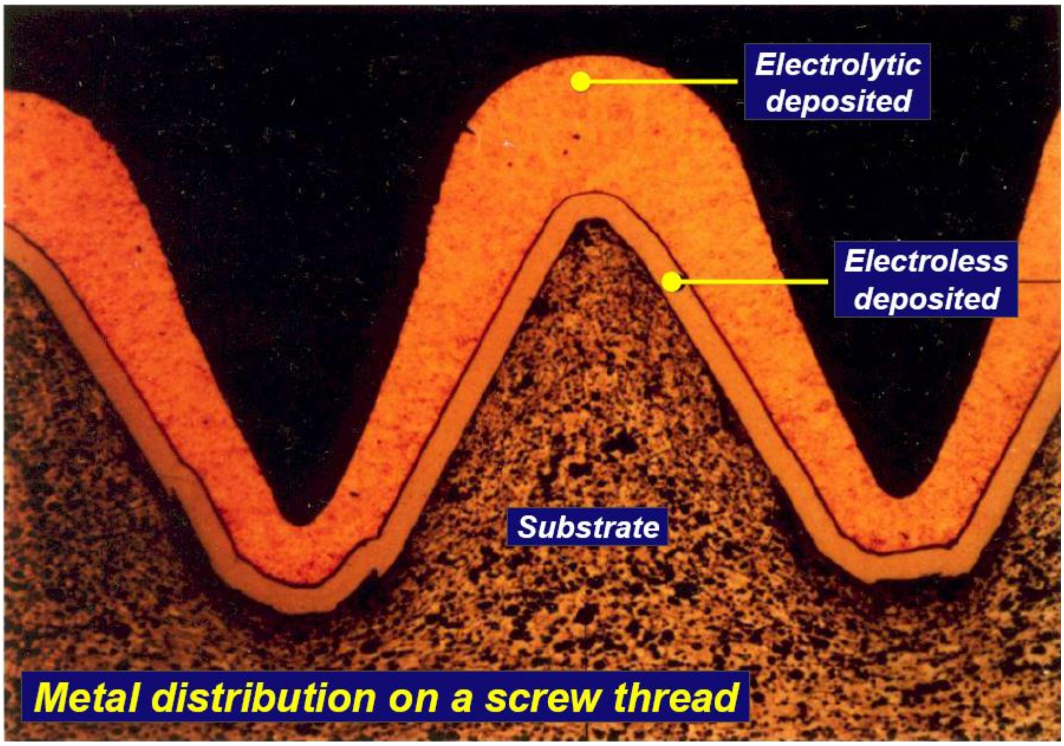

Figure 1.2. Metal distribution of electrolytic vs. electroless deposits on a screw thread.

1.2.2.1. Advantages and disadvantages of electroplating

1.2.2.1.1. Advantages of electroplating

Advantages of electroplating are listed below.

- Very wide range of metals and metal alloys can be deposited
- Wide range of substrates can be electroplated – both metals and non-metals
- Different coatings can be deposited consecutively e.g. copper plus nickel plus gold, palladium or chromium on die-castings or plastics.

- Providing a correct pre-treatment a good adhesion of coating to substrate is possible
- Coating thickness can vary from less than 0,1 µm to several mm
- Several surface structures can be produced, from dull to satin look to mirror bright.

1.2.2.1.2. Disadvantages of Electroplating

Disadvantages of electroplating are listed below.

- Often hazardous chemicals are used which can pose health and safety problems to people and the environment.
- Many very effective substances are banned for environmentally or health reasons. Replacements are often less effective or more expensive, e.g. cyanides.
- Necessary process sequences can influence properties of substrates negative, e.g. brittleness due to hydrogen adsorption.
- In electroplating it is often not to see what is actual happening. Electroplating is kind of “Black Art” in some areas of activity.

1.2.3. Electrolytic zinc coating

Historically the use of zinc as thin corrosion protection layer dates back to the middle of the 19. century. With the introduction of the dynamo by Werner von Siemens in 1867 the durability for mass production of zinc plating was established. First patents for zinc electrolytes date back also in these times. Industrial significance could be achieved from the 1920 on with the introduction of cyanide alkaline electrolytes. These processes dominated the next period with robustness and easy handling. The development went further via acid electrolytes and cyanide free alkaline electrolytes[1].

Presently the main driver for further developments in corrosion protection is the automotive industry with increased quality demands and extended warranty periods but the classical zinc layers reach their limit. The consequence was the introduction of higher performing zinc alloy surfaces. In addition to zinc-iron, nowadays zinc-nickel deposits dominate the zinc alloy market. The vast majority of goods are plated from alkaline based electrolytes. Up to now, acid based alternatives could not convince, since the properties of the deposit from these first generation electrolytes were outperformed by these plated from alkaline solutions. Now a new generation of ammonia free acid zinc nickel solutions is available and latest research and development has led to a reliable and equal performing alternative to commonly used alkaline solutions.

Zinc and zinc coatings have been investigated for various technological applications in recent years due to their structural properties, especially electronegativity difference between iron and zinc and corrosion resistance. Zinc coatings with different chemical compositions and applicable properties are synthesized in amazing diversity [1]. Some of the zinc coatings are alkaline, acidic, cyanide zinc coatings and alkaline, acidic zinc nickel alloy coatings and alkaline zinc iron alloy coatings with different corrosion resistances, hardness values, deformation behaivours, magnetic and optical properties. Zinc coatings have good adhesion and shapening properties and they do not decrease the structural and mechanical properties of the base material during coating [2,3].
Raised quality requirements coming along with extended warranty periods call for improved corrosion protection quality in car manufacture, generally. Regarding zones in the car experiencing thermal stress, like under-hood areas where traditional pure zinc coatings have limited performance which moreover the significance of coatings.

1.2.4. Electrolytic Zinc Nickel Alloy Coating

Zinc-nickel coatings with a nickel incorporation of 12 to 16 % provide superior corrosion protection to red rust compared to pure zinc coatings due to the deposition of g-phase zinc-nickel alloy (**Figure 1.3**). The corrosion protection is stable even when heat is applied. Zinc nickel electrolytes are all composed to plate highest amounts of pure g-phase zinc-nickel alloy providing the best corrosion protection. XRD and SEM investigations as well as FIB investigations have been undertaken to achieve best possible deposit properties over the complete current density range. As a result, the corrosion protection rates achieved in neutral salt spray test clearly exceed the demands of the automotive industry.

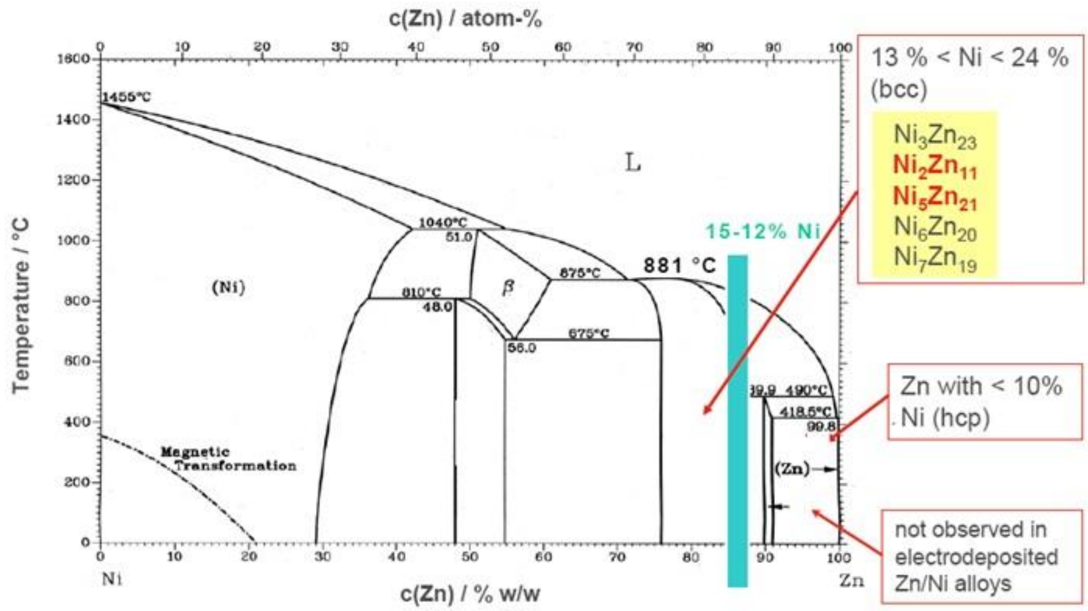

Figure 1.3. Phase diagram for zinc and nickel.

A diversity of alkaline based zinc-nickel electrolytes is available and well established in the industry already for a diversity of applications. Until now acid electrolytes did not play an important role due to the inhomogeneous nickel incorporation und therefore inferior corrosion protection performance. With the new generation of acid based ammonia free electrolyte this situation has changed. Homogenous deposition of a gamma phase Ni_2Zn_{11} zinc-nickel alloy giving highest corrosion protection performance is now available from acidic electrolytes, as well. It enables to coat casting parts with high corrosion resistance.

1.2.4.1. Advantages of zinc nickel over zinc and zinc iron

Unquestionably, the excellent cathodic corrosion protection properties of zinc nickel are the most important benefit over non-alloy zinc layers. Five main advantages of zinc nickel surfaces can be named by

- Best corrosion protection against red rust
- Less voluminous white rust appearance
- Low contact corrosion in contact to aluminum

- High wear resistance
- Good temperature stability

Figure 1.4 shows the result of an investigation with different zinc and zinc alloy coatings. Panels with different thicknesses have been plated without further post treatment. After subjecting to neutral salt spray test (ASTM B-117 NSS) the time to red rust and the resulting corrosion speed was assessed. With more than 700 h to red rust zinc nickel coatings with a nickel incorporation of 14 %, followed by low alloy coatings with 6.5 % nickel and 220 h to RR, these alloys clearly exceed the performance of pure zinc with 150 h at an average plating thickness of 8 µm (**Figure 1.5**).

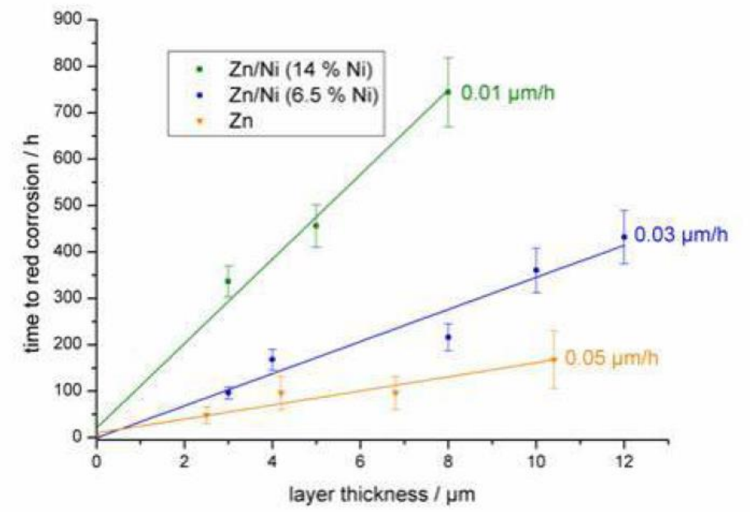

Figure 1.4. Neutral salt spray test according to ASTM B117 on panels with different zinc and zinc alloy coatings and varying thicknesses.

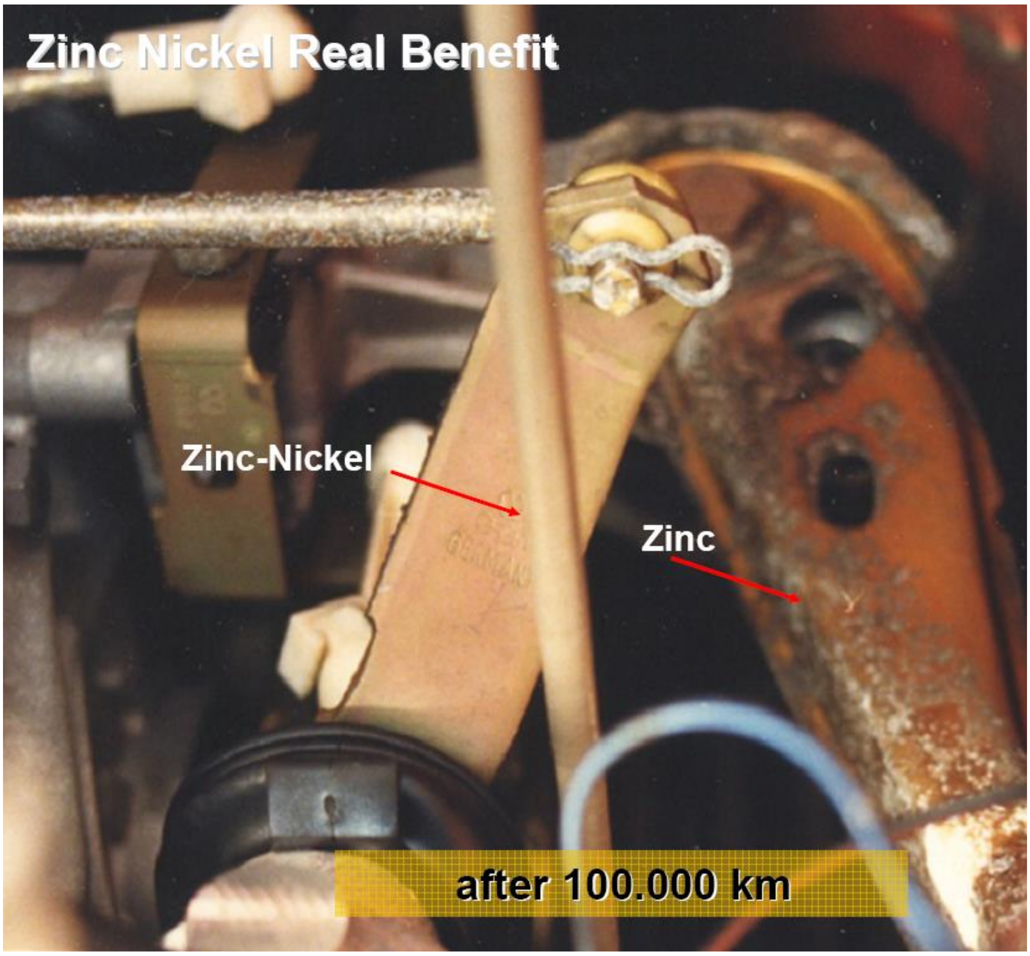

Figure 1.5. Corrosion resistance of zinc vs. zinc nickel deposit on assembled automotive parts after 100.000 km usage.

Further investigations in the structure of zinc nickel coatings with x-ray diffraction have shown that the corrosion protection depends on the formation of gamma phase zinc nickel. In Figure 1.6, it becomes evident, that the presence of gamma phase correlates to the corrosion protection of zinc nickel surfaces. The graph shows, the higher the proportion of Ni_2Zn_{11}, the better the corrosion protection in relation to zinc. Maximum concentration of gamma phase structure in the layer can be achieved with a nickel incorporation of 12 – 16 % which also offers the best sacrificial corrosion protection for the substrate.

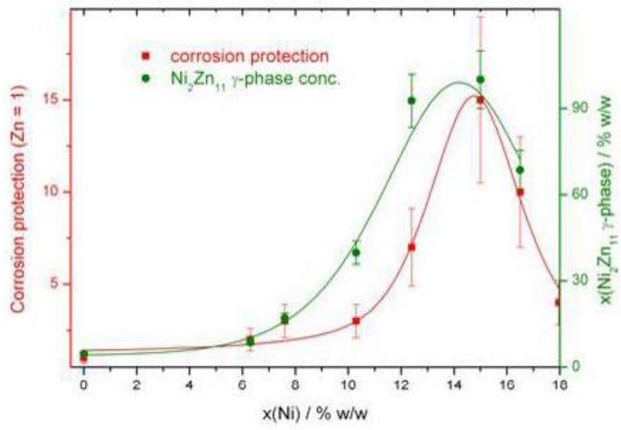

Figure 1.6. Corrosion protection relative to pure zinc and proportion of gamma phase zinc nickel as function of nickel content in the layer.

The homogenous distribution of this gamma phase deposition is crucial for successfully applying cathodic corrosion protection. Alkaline electrolytes ensure very homogenous nickel incorporation. As a result a homogenous gamma-phase deposition over a broad range of current densities can be observed. Due to this, still alkaline electrolytes are state-of-the-art. Nevertheless it is important to keep in mind that for some applications these electrolytes have also their limits. When talking about the plating of bulk articles, for example fasteners, especially of hardened steel material often difficulties with reliable coverage lead to rework. For materials as cast iron it is commonly known that the use of acid based electrolytes have their benefits to achieve good plating results. The higher current efficiency of acidic solutions allows a faster nucleation and therefore a better coverage of these substrates.

Zinc nickel is that inclusion of nickel modifies corrosion potential; zinc nickel alloy more noble than zinc.

- Sacrificial protection to steel maintained and corrodes at a lower rate. Same thickness protects steel longer than single zinc.
- Developed in the past to be a replacement for cadmium after the ban of cadmium.
- Renewed interest in Zinc Nickel deposition technology due to increased or changed industrial demands to deposit properties that are not attainable with single metal coatings.

2. MATERIAL AND METHOD

For acidic and alkaline zinc nickel coating investigations, approx. 5 x 5 cm^2 casting and zamak panels were plated in the alkaline and acid zinc nickel bath solution at current densities of 0.7 and 2.5 A/dm^2.

The acidic zinc nickel bath solution was made up with values in the Table 2.1 and acid zinc nickel electrolyte working parameters in Table 2.2.

Table 2.1. Acidic zinc nickel bath make up.

Make up of 1 liter	ml/l	g/l
Water	Approx. 900	Approx. 900
Potassium chloride	---	200 g/l
Zinc chloride	---	40 g/l
Boric acid	---	20 g/l
Nickel chloride x 6 H_2O	---	120 g/l
Basic solution	10 m/l	---
Brightener	20 m/l	---
Buffer	---	60 g/l
Wetting agent	20 m/l	

Basic solution, complexing additive required for proper bath performance

Brightener used for the whole current density area.

Buffer is to prevent burnings in high current densities.

Wetting agent, used for the low to medium current density area for a better leveling.

- Fill the tank with 500 ml warm water (recommended at approx. 50 °C).
- Add 20 g of boric acid, mix to dissolve.
- Add 40 g of zinc chloride, mix to dissolve.
- Add 120 g of nickel chloride, mix to dissolve.
- Add 200 g of potassium chloride, mix to dissolve.
- Add 60 g of buffer, mix to dissolve.
- Start filter pump and circulate solution.

- After the solution has reached the operating temperature, add 10 ml basic solution
- Place anode material onto the anode bars and adjust solution the level to the final working volume and do some dummy plating for 2 Ah/l.
- Then add 20 ml brightener and 20 ml wetting agent.
- The solution pH should be checked and adjusted as required.

Table 2.2. Acidic zinc nickel electrolyte working parameters.

Temperature	Rack: 35 (30 – 45) °C
Ph	5.5 (5.2 – 5.7) (Lyphan(TM)-Paper range 4.3 - 6.1) pH should be monitored and can be adjusted by addition of hydrochloric acid or potassium hydroxide solution.
Voltage	Bath should not be operated if voltage is higher than 8 V.
Current density	For rack plating: 2 A/dm2 (0.5 - 3 A/dm2)
Rate of deposition	For rack plating: approx. 0.6 – 0.8 µm in 1 min at 3 A/dm2

The alkaline zinc nickel bath solution was made up with values in the Table 2.3 and alkaline zinc nickel electrolyte working parameters in Table 2.4.

Table 2.3. Alkaline zinc nickel electrolyte make up.

Make up of 1 liter	ml/l	g/l
Water	Approx. 900	Approx. 900
Sodium hydroxide	---	130 g/l
Zinc oxide	---	14 g/l
Carrier	87	90
Brightener	1	1
Nikel solution	3	3

Carrier complexing additives required in correct ratio for proper bath performance.

Brightener used for the medium to low current density area.

Nickel solution is nickel replenishment additive.

- Fill the tank with 350 ml water.
- With solution temperature of the base electrolyte below 27°C, make addition of the specified quantities of carrier additives in the following order:
- Whilst stirring brightener and nickel solution.
- Place anode material onto the anode bars and adjust solution level to the final working volume.
- Adjust solution temperature within working range, electrolyte is now ready for use.

Casting and zamak parts were coated about 8 μm in the both alkaline and acidic zinc nickel bathes then some parts were detached to investigate as originally. Other parts were put in the neutral salt spray test cabin until 240 and 480 hour according to ASTM B-117.

The coated parts with 2.5 A/dm^2 were investigated by X-ray fluorescence (XRF), X-ray diffraction (XRD), focued ion beam (FIB), optical microscope and scanning electron miscope (SEM).

The morphology of the coatings was examined by scanning electron microscopy. Cross sections of these panels were made by a focused ion beam instrument to investigate the texture of the deposits. The zinc nickel alloy phase composition of these panels was measured by X-ray diffraction. The thickness and the zinc nickel composition of the coatings was determined using X-ray fluorescence. The corrosion protection was tested in neutral salt spray solution test according to ASTM B-117.

3. RESULTS AND DISCUSSION

3.1. Surface Morphology of Zinc Nickel Alloy Deposits

Acidic zinc nickel is vital to prove that the deposit achieved from the new electrolyte has the same properties as the deposit plated from alkaline zinc nickel solutions. A number of investigations have been conducted to compare both deposits.

Samples were plated at 0.7 A/dm² and 2.5 A/dm² and examined with a scanning electron microscope. Expecting a globular surface morphology resulting from an alkaline zinc nickel electrolyte Figure 3.1 shows that deposits from both types of electrolytes show similar globular morphology for both current densities tested.

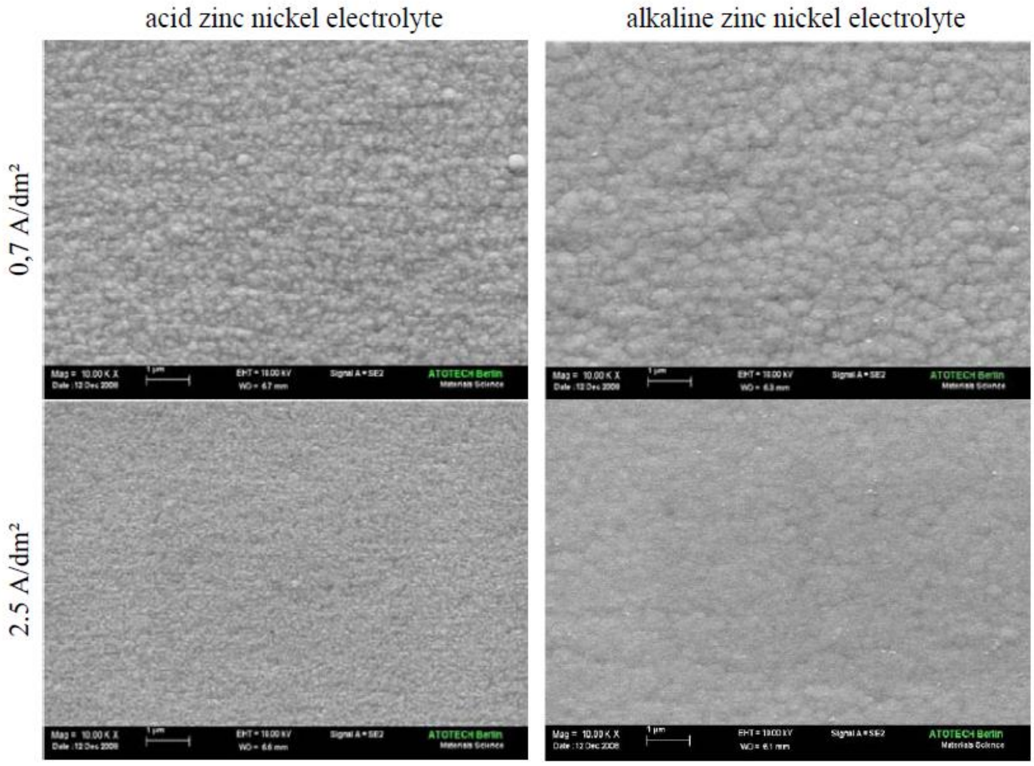

Figure 3.1. SEM cross section image of surface morpholgy comparing zinc nickel alloy deposit from acid and alkaline electrolytes.

Further to the surface morphology also the crystallite texture was determined by means of focused ion beam (FIB). As the surface morphology indicates already, Figure 3.2 confirms the same field oriented columnar crystallite texture. This is a typical characterization for the zinc nickel structure.

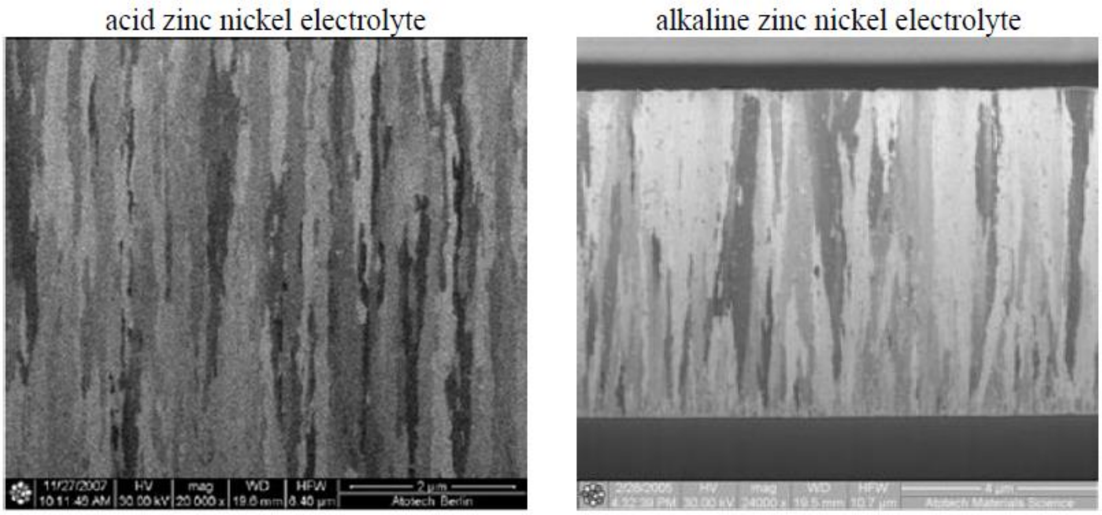

Figure 3.2. FIB cross section image of zinc nickel coating form acid and alkaline electrolytes.

With the deposit of gamma phase structure being essential for the corrosion protection performance of the parts plated with zinc nickel, x-ray diffraction patterns have been recorded to analyze the phase composition of layers from acidic and alkaline zinc nickel electrolytes.

Figure is the XRD obtained from a sample plated from an alkaline electrolyte, which is typically used for rack application, at 2.5 A/dm² with a thickness of 8 µm.

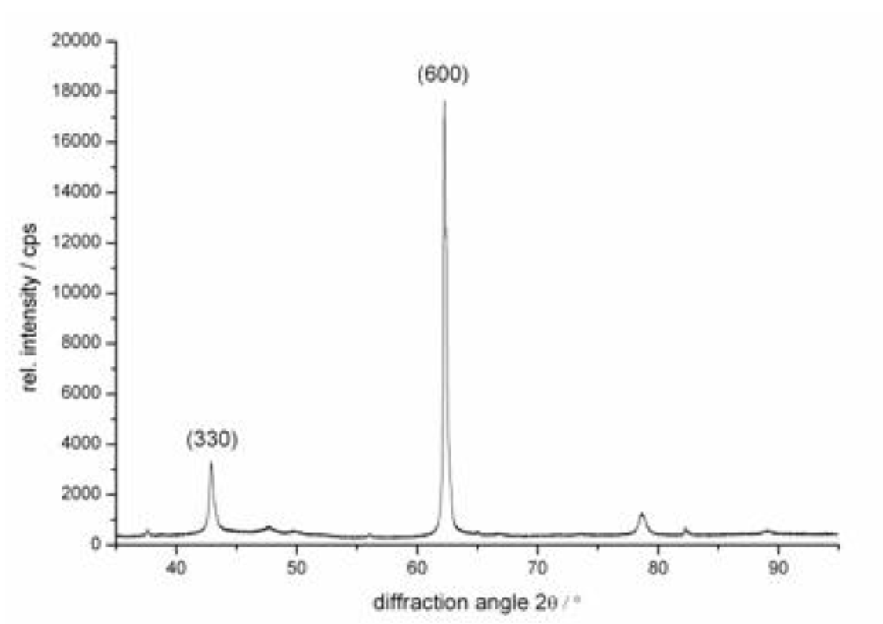

Figure 3.3. XRD Scan of a zinc nickel deposit from an alkaline based electrolyte.

When compared the XRD scan (**Figure 3.3**) of the surface of sample plated from a production proven electrolyte (2.5 A/dm², 8 µm) with the scan of the alkaline electrolyte (**Figure 3.4**), it can be seen, that both patterns show the presence of pure gamma phase structure. Both patterns are almost congruent.

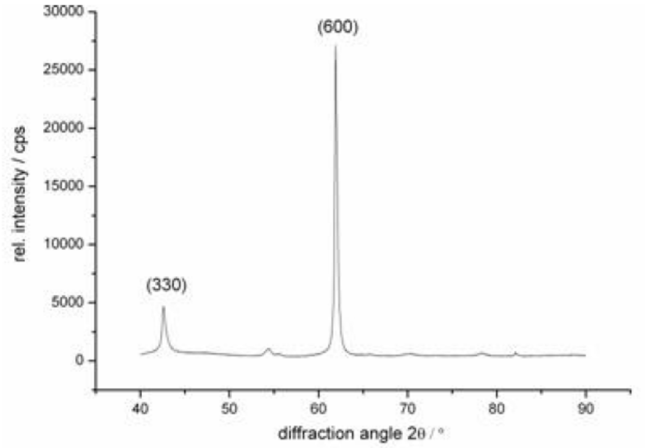

Figure 3.4. XRD Scan of a zinc nickel deposit from an acid based zinc nickel electrolyte.

3.2. Zinc Nickel Alloy Deposits Investigated by Optic Microscope and SEM

Alkaline and acidic zinc nickel coated casting and zamak parts were investigated by optical microscope and scanning electron microscope after 240 h and 480 h neutral salt spray test and also coating without neutral salt spray test.

3.2.1. Alkaline Zinc Nickel Coating on Cast Parts

Alkaline zinc nickel coated casting parts were investigated by optic microscope and scanning electron microscope after 240 h and 480 h neutral salt spray test and also coating without neutral salt spray test. Then images are commented.

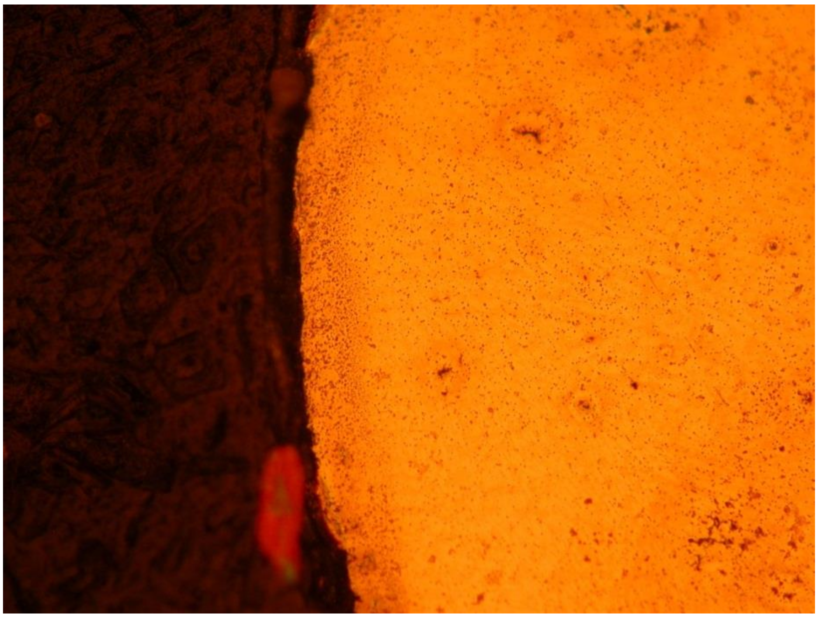

Figure 3.5. Optical microscope cross section image of alkaline zinc nickel coating on the casting part (2000x magnification).

Figure 3.6. Optical microscope cross section image of alkaline zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (1000x magnification).

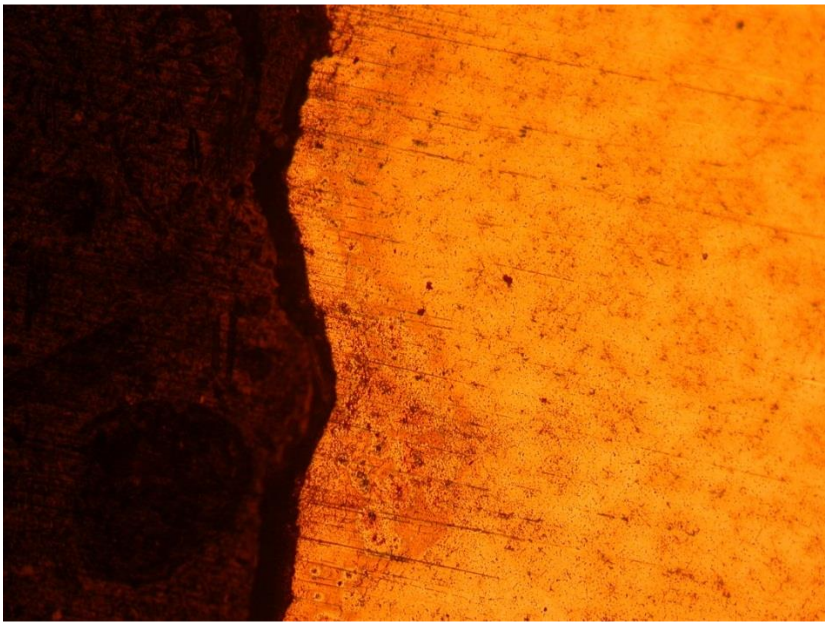

Figure 3.7. Optical microscope cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

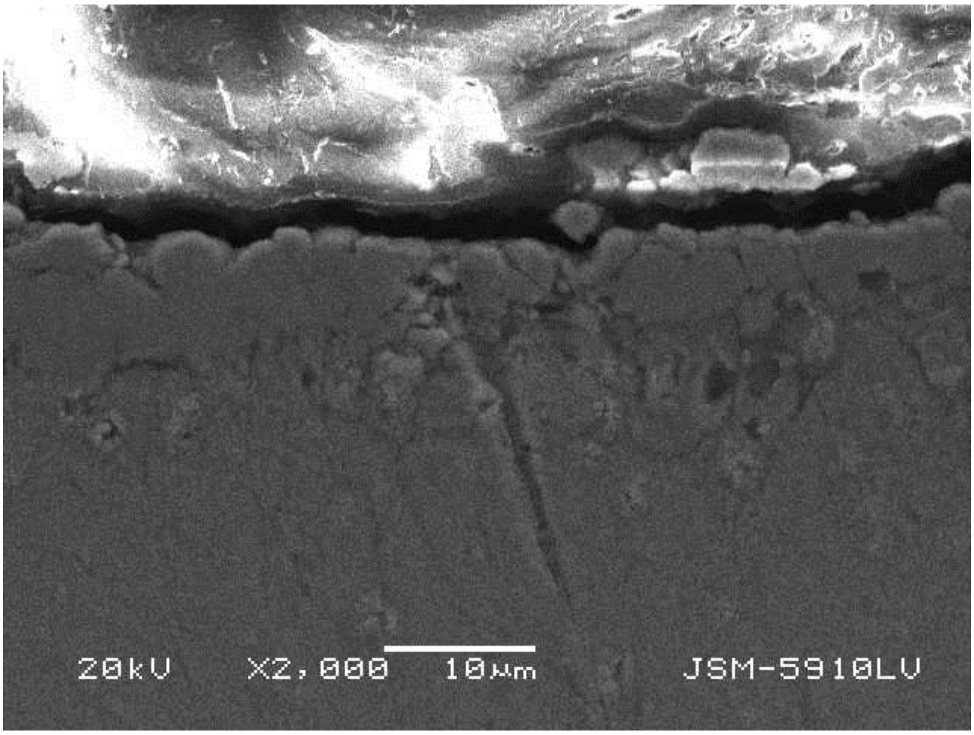

Figure 3.8. SEM cross section image of alkaline zinc nickel coating on the zamak part (2000x magnification).

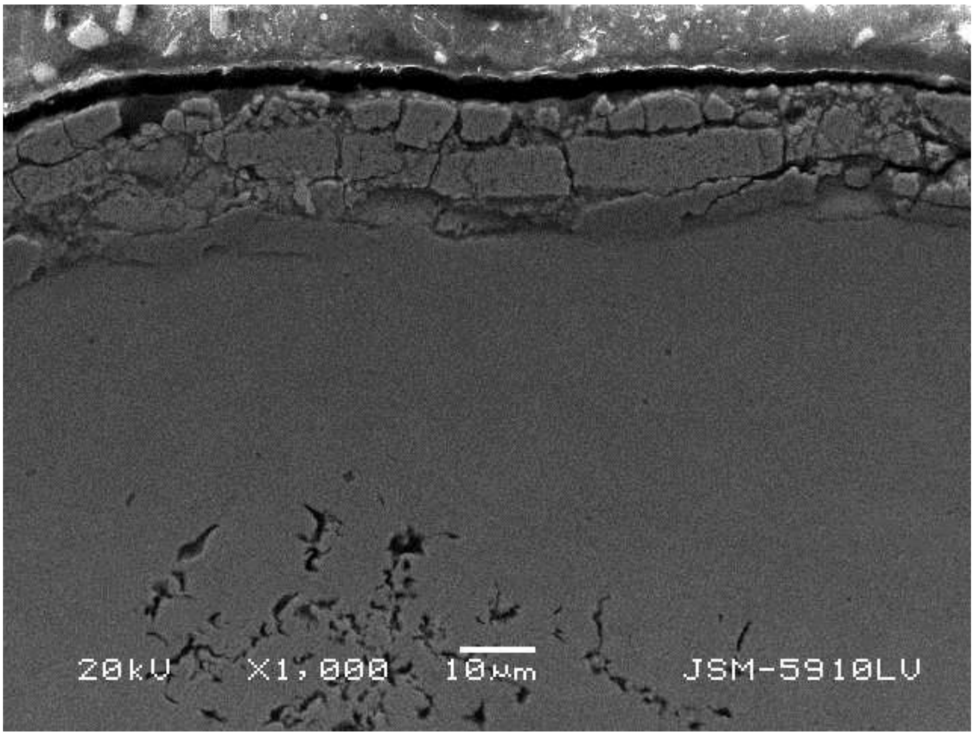

Figure 3.9. SEM cross section image of alkaline zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (1000x magnification).

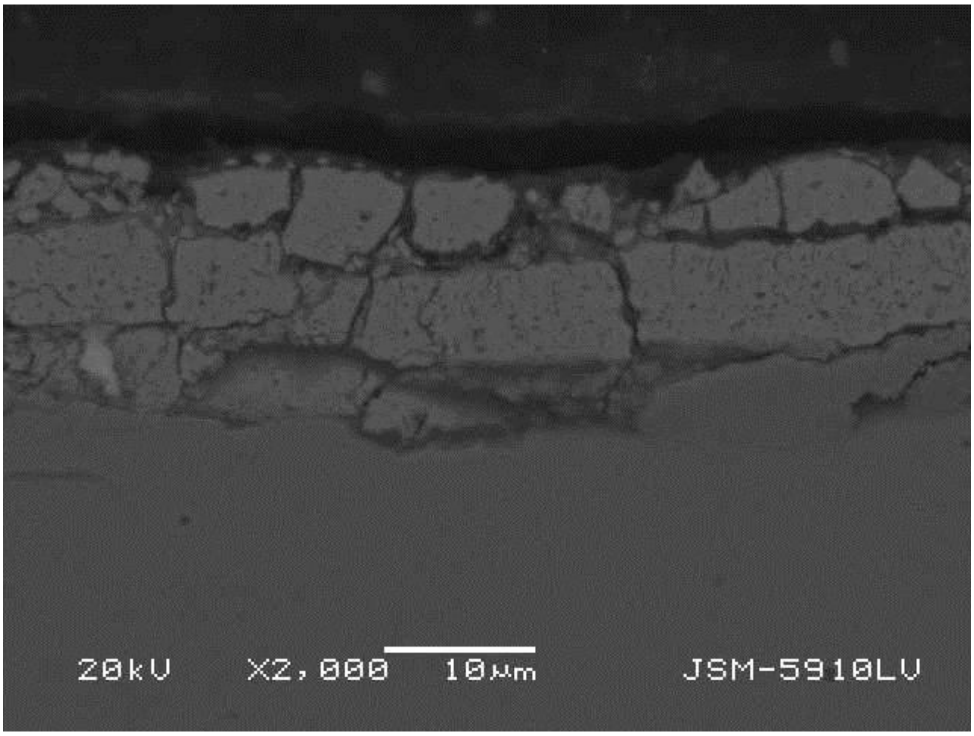

Figure 3.10. SEM cross section image of alkaline zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

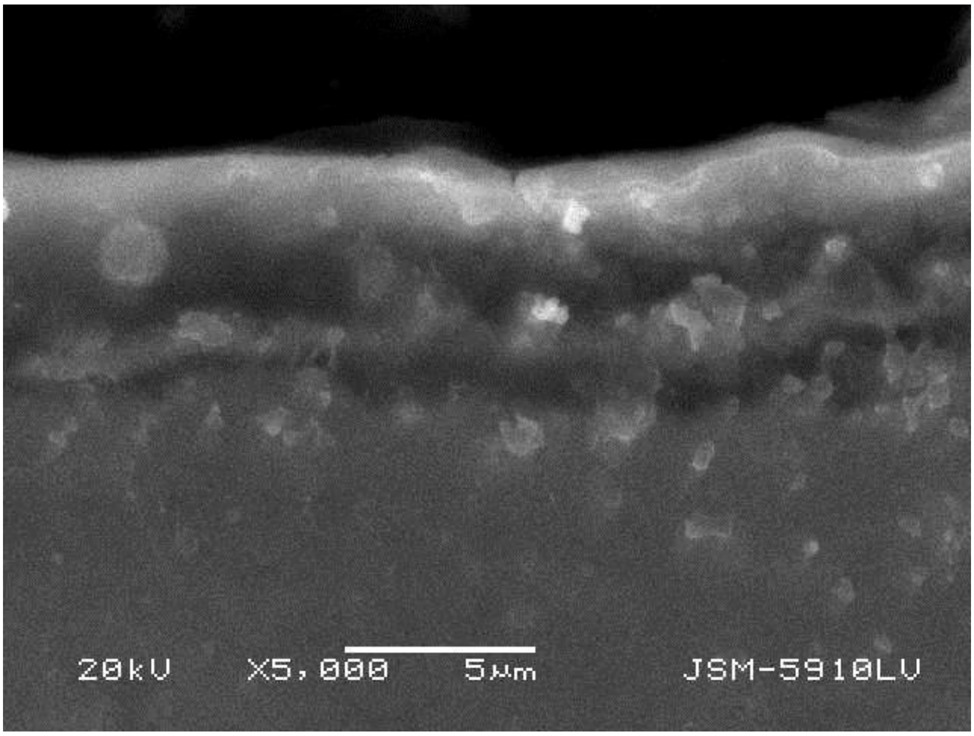

Figure 3.11. SEM cross section image of alkaline zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (5000x magnification).

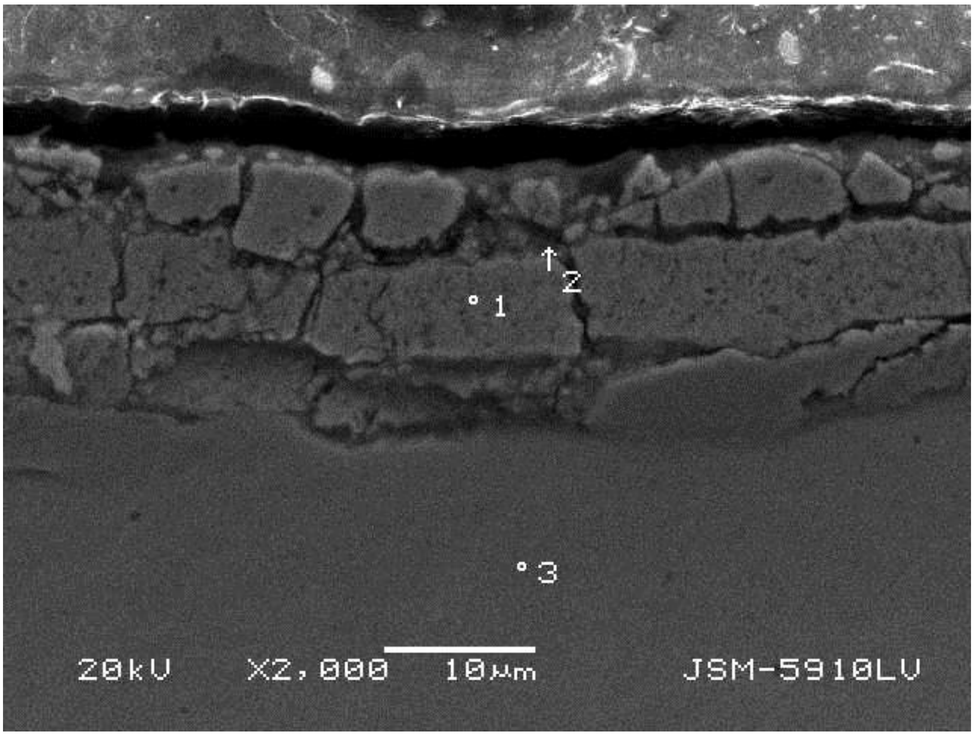

Figure 3.12. SEM - EDS cross section image of alkaline zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

Elements of number 1 as marked on Figure 3.13 was explained on the Table 3.1 and showed as graph on Figure 3.14.

Table 3.1. All elements on Figure 3.14 analyzed for point 1 on Figure 3.13.

Spectrum	O	Fe	Ni	Zn	Total
Spectrum 1	4.46	1.86	11.86	81.82	100.00
Max.	4.46	1.86	11.86	81.82	
Min.	2.00	1.41	11.72	76.25	

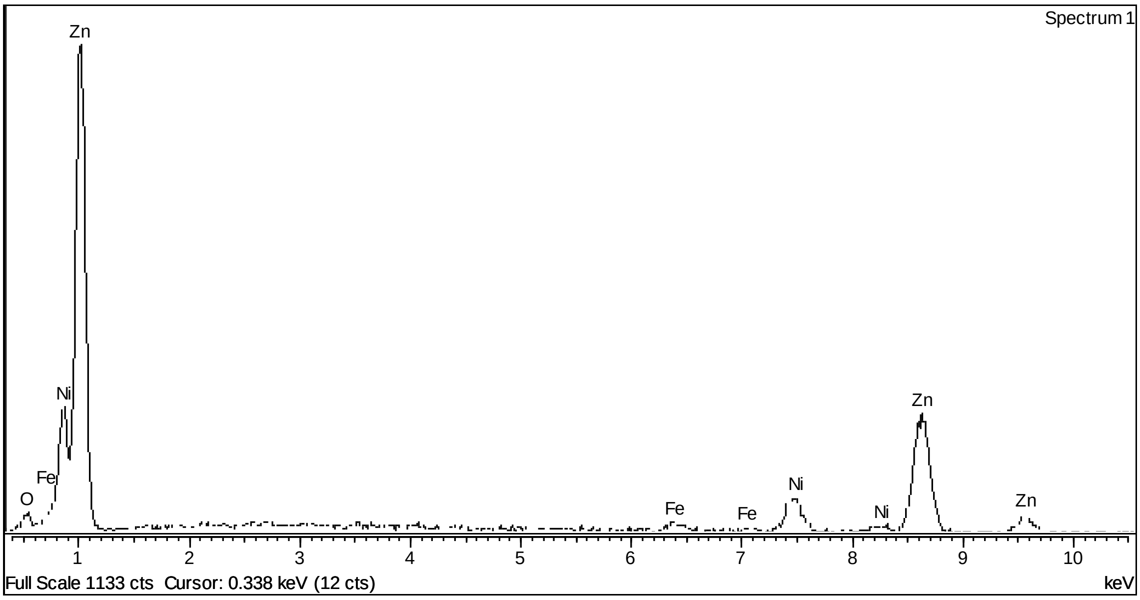

Figure 3.13. All values in Table 3.1 are as weight percent.

Elements of number 2 as marked on Figure 3.13 was explained on the Table 3.2 and showed as graph on Figure 3.14.

Table 3.2. All elements on Figure 3.14 analyzed for point 2 on Figure 3.71.

Spectrum	O	Cl	Ca	Fe	Ni	Zn	Total
Spectrum 1	23.57	1.42	0.52	1.12	4.80	68.57	100.00
Max.	23.57	1.42	0.52	1.12	4.80	68.57	
Min.	23.57	1.42	0.52	1.12	4.80	68.57	

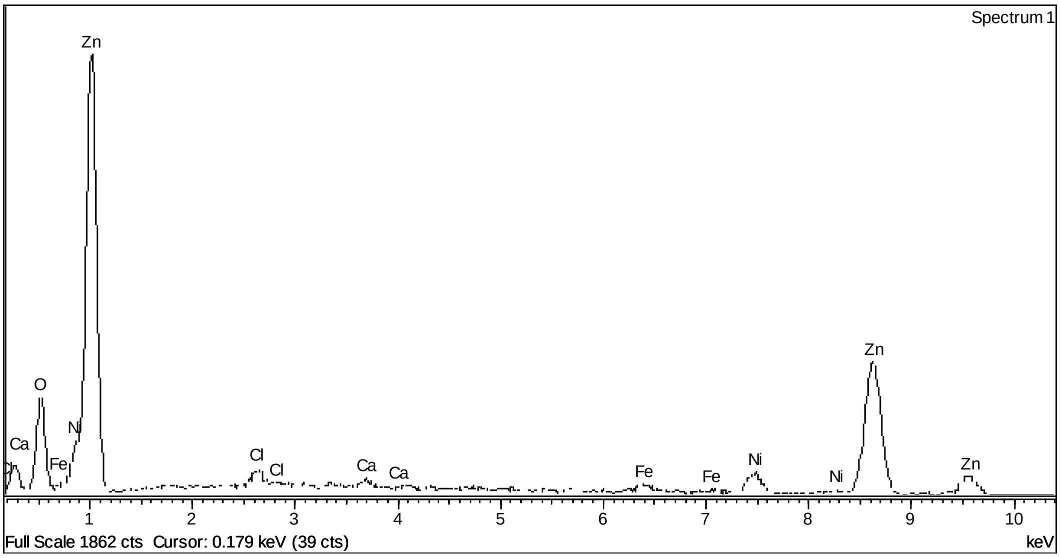

Figure 3.14. All values in Table 3.2 are as weight percent.

Elements of number 3 as marked on Figure 3.13 were explained on the Table 3.3 and showed as graph on Figure 3.15.

Table 3.3. All elements Figure 3.15 analyzed for point 3 on Figure 3.73.

Spectrum	Si	Mn	Fe	Total
Spectrum 1	2.99	0.63	96.38	100.00
Max.	2.99	0.63	96.38	
Min.	2.99	0.63	96.38	

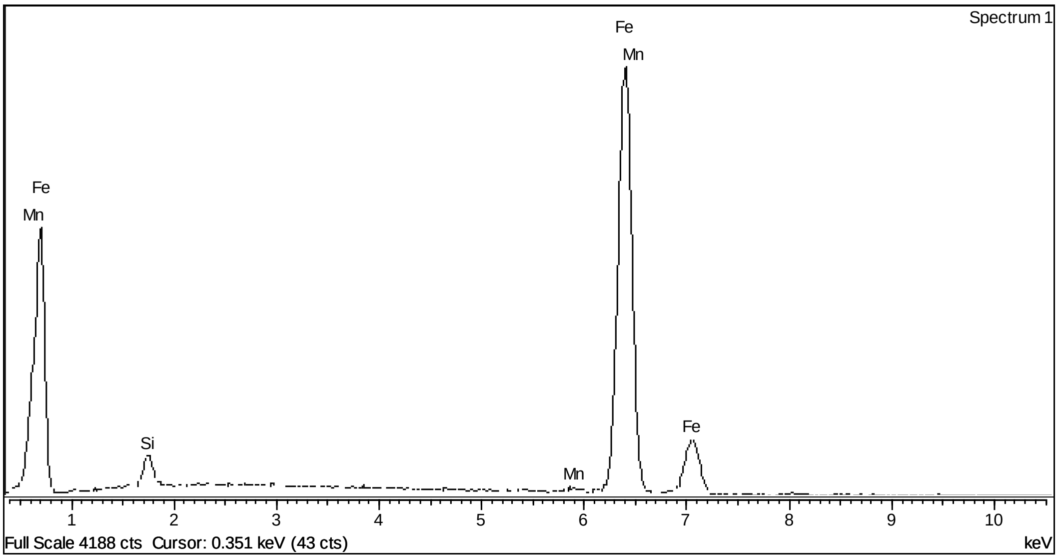

Figure 3.15. All values in Table 3.3 are as weight percent.

We analyzed all elements at 1, 2 and 3 points on Figure 3.13 by EDS. Analyzed sample exposed 240 h ASTM B 117 neutral salt spray test. Point 1 is the middle of the coating and indicated % 4,46 O, % 1,86 Fe, % 11,86 Ni and % 81,82 Zn which show that it did not expose corrosion. Point 2 is corner of the coating analyzed as % 23,57 O, % 1,42 Cl, % 0,52 Ca, % 1,12 Fe, % 4,80 Ni and % 68,57 Zn. This element levels show that no corrosion occurred. Point 3 is middle of the casting substrate and analyzed as % 2,99 Si, % 0,63 Mn and % 96,38 Fe indicating that no corrosion formed.

Optical microscope and SEM results of alkaline zinc nickel coating on the casting parts of original samples before salt spray test showed smooth and permanent coating. After 240 h salt spray test, black spot areas were seen at optical microscope images and also cracks and cleavages were detected in SEM images. 480 h salt spray test showed larger black spots areas in optical microscope images and larger distances between cracks and cleavages in the SEM images. Corrosion damages coating and struggle to find a way to reach base metal. EDS data supports this comment.

3.2.2. Alkaline Zinc Nickel Coating on Zamak Parts

Alkaline zinc nickel coated zamak parts were investigated by optical microscope and scanning electron microscope after 240 h and 480 h neutral salt spray test and also coating without neutral salt spray test.

Figure 3.16. Optical microscope cross section image of alkaline zinc nickel coating on the zamak part (1000x magnification).

Figure 3.17. Optical microscope cross section image of alkaline zinc nickel coating on the zamak part (2000x magnification).

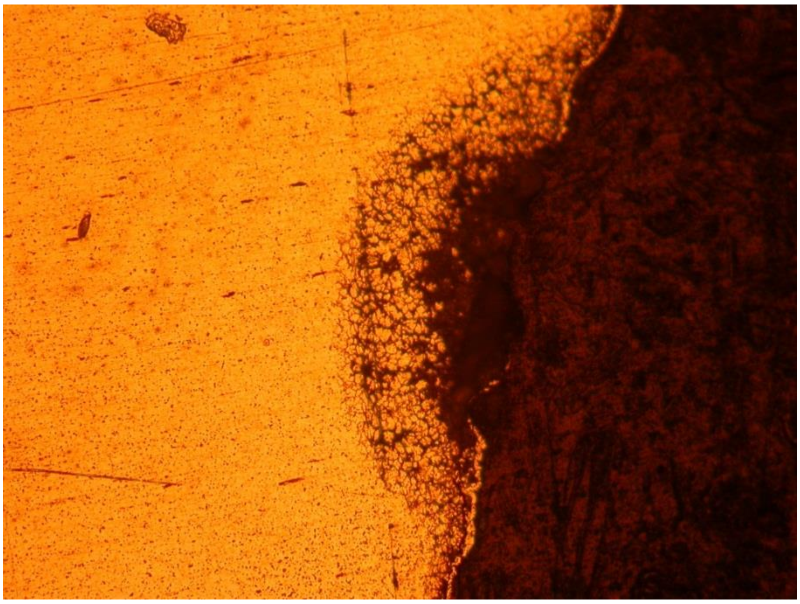

Figure 3.18 Optical microscope cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (1000x magnification).

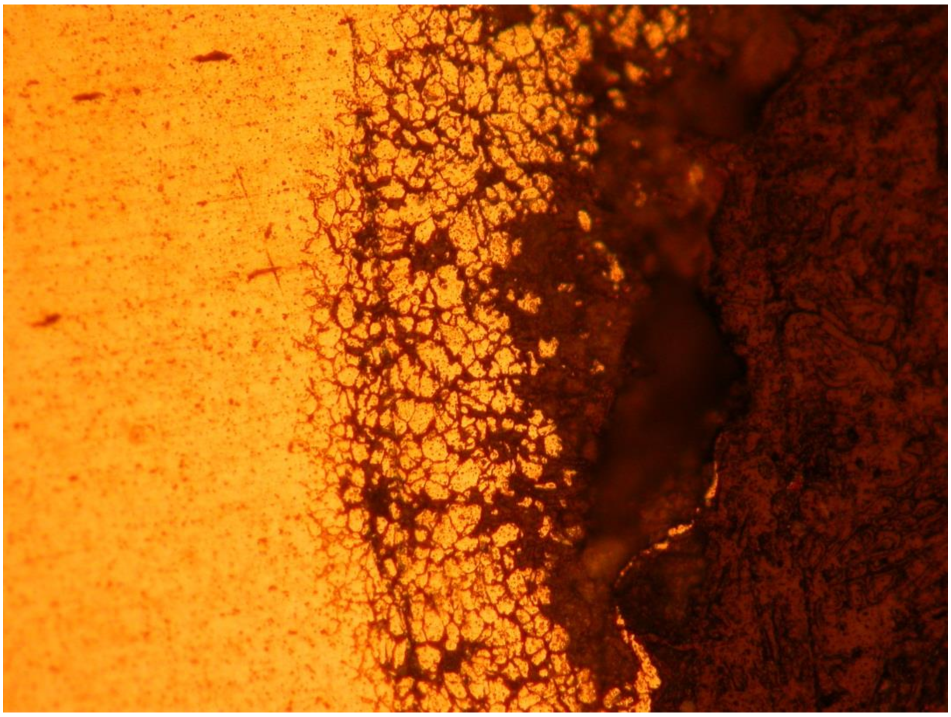

Figure 3.19. Optical microscope cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

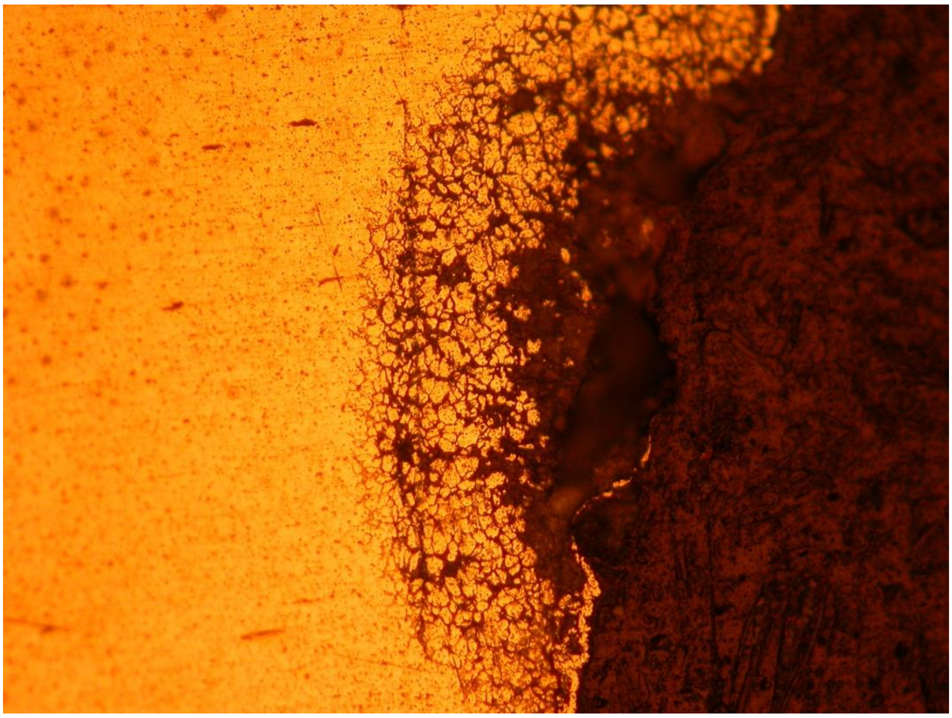

Figure 3.20. Optical microscope cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (5000x magnification).

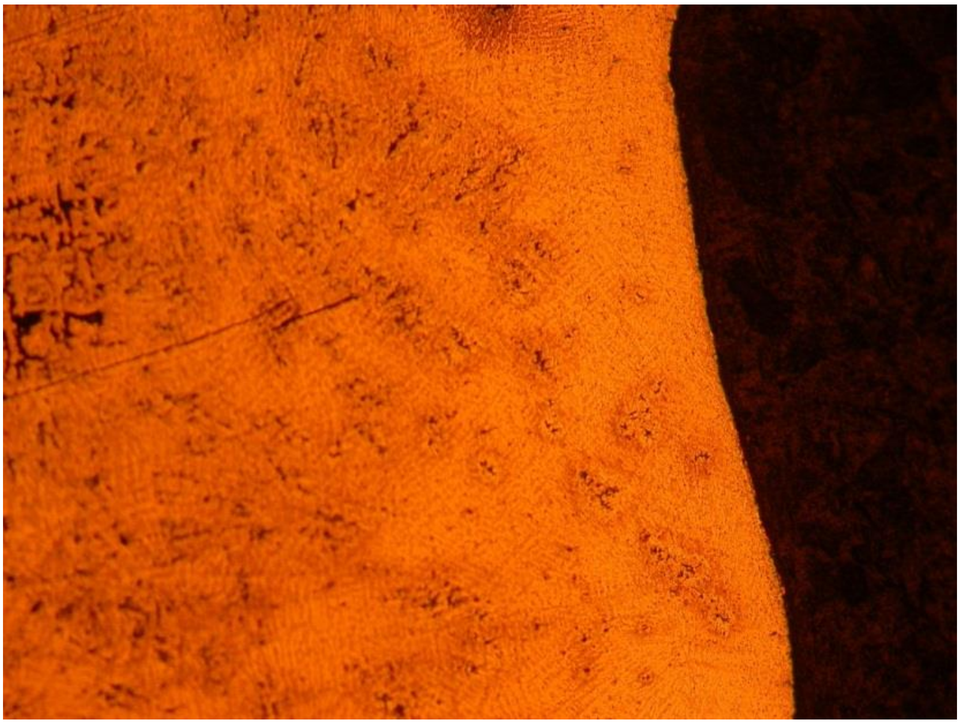

Figure 3.21. Optical microscope cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (1000x magnification).

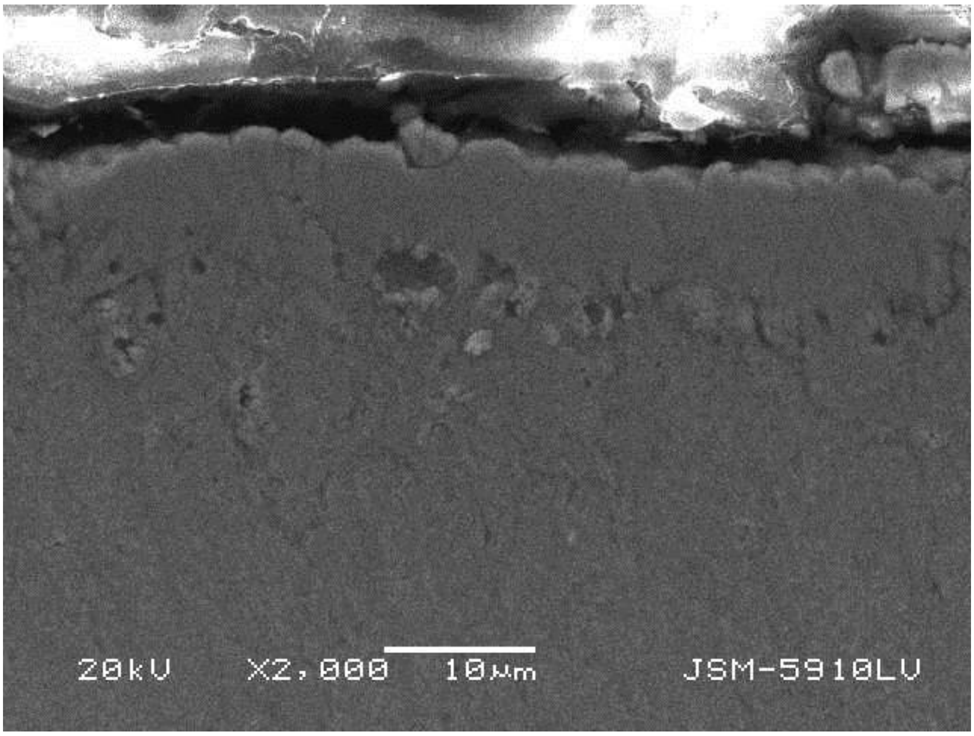

Figure 3.22. SEM cross section image of alkaline zinc nickel coating on the zamak part (2000x magnification).

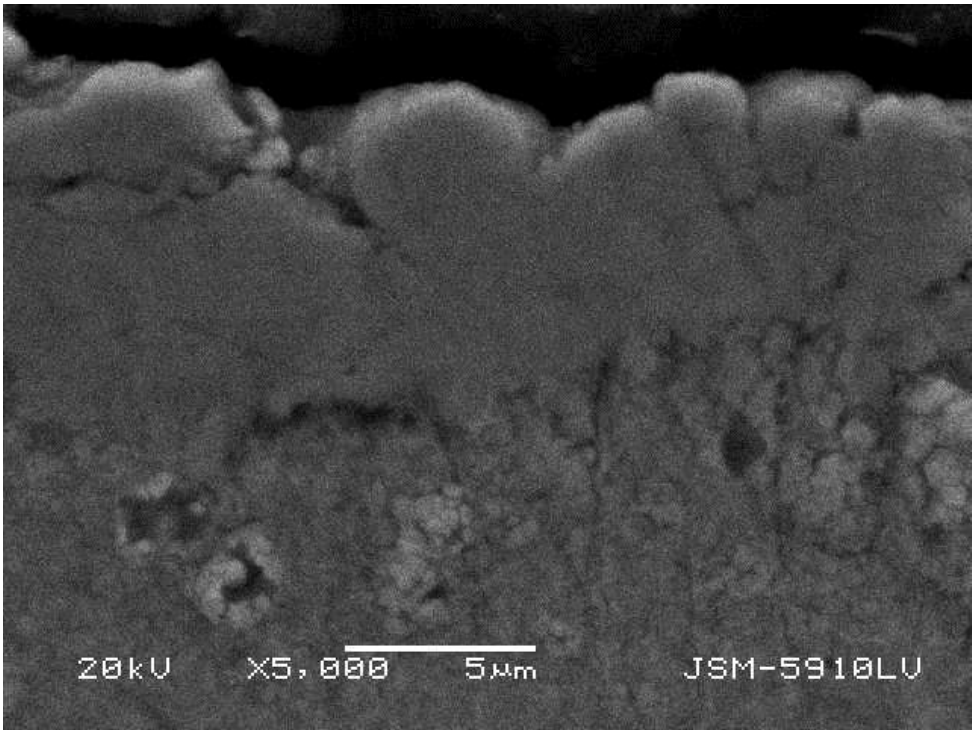

Figure 3.23. SEM cross section image of alkaline zinc nickel coating on the zamak part (5000x magnification).

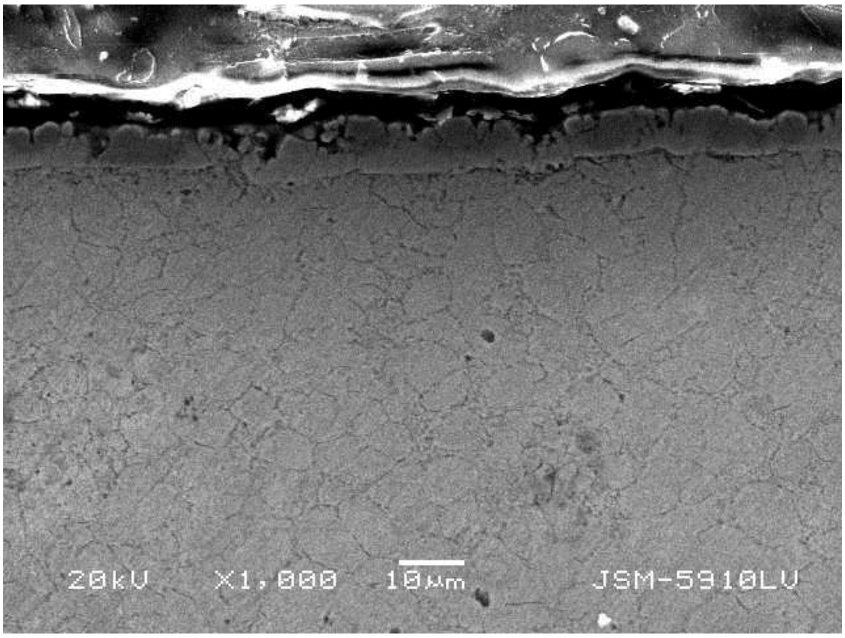

Figure 3.24. SEM cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (1000x magnification).

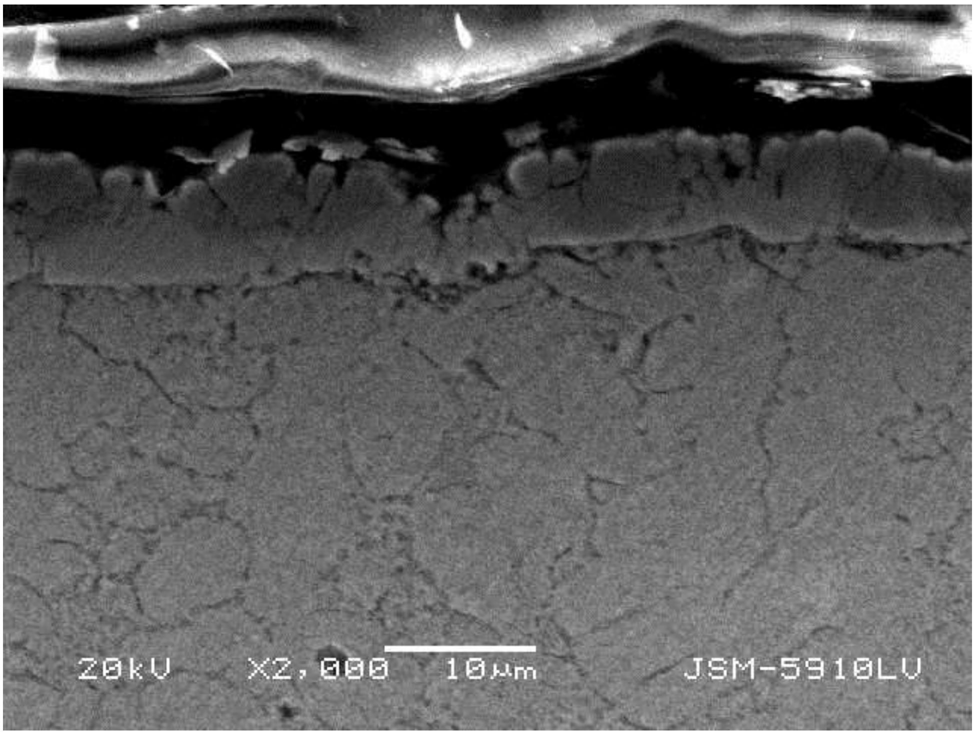

Figure 3.25. SEM cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

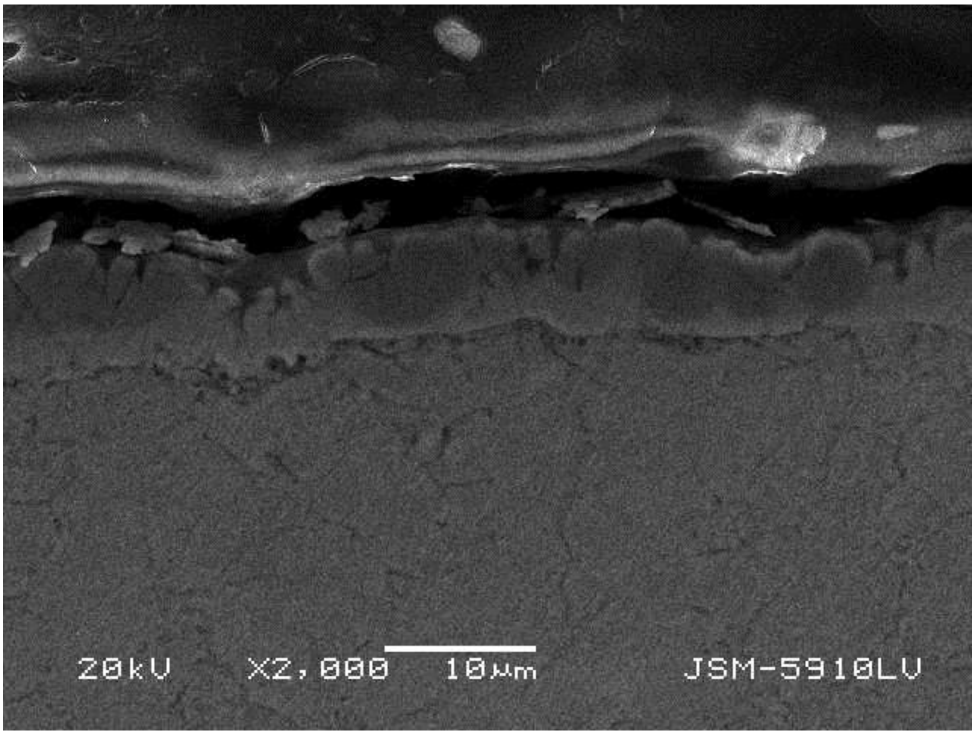

Figure 3.26. SEM cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

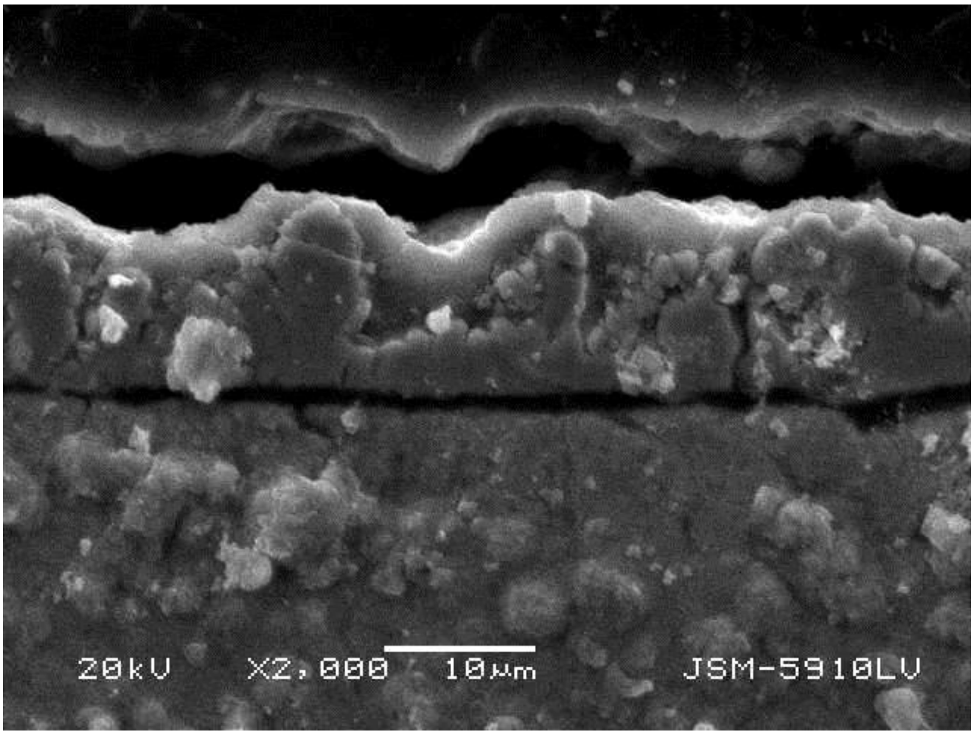

Figure 3.27. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

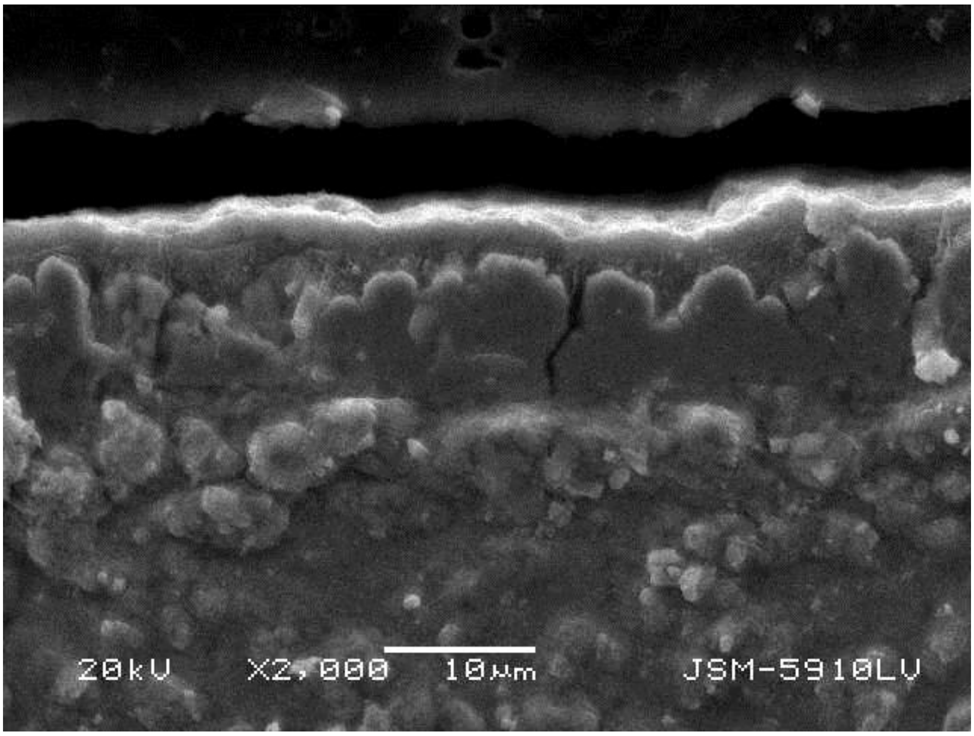

Figure 3.28. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

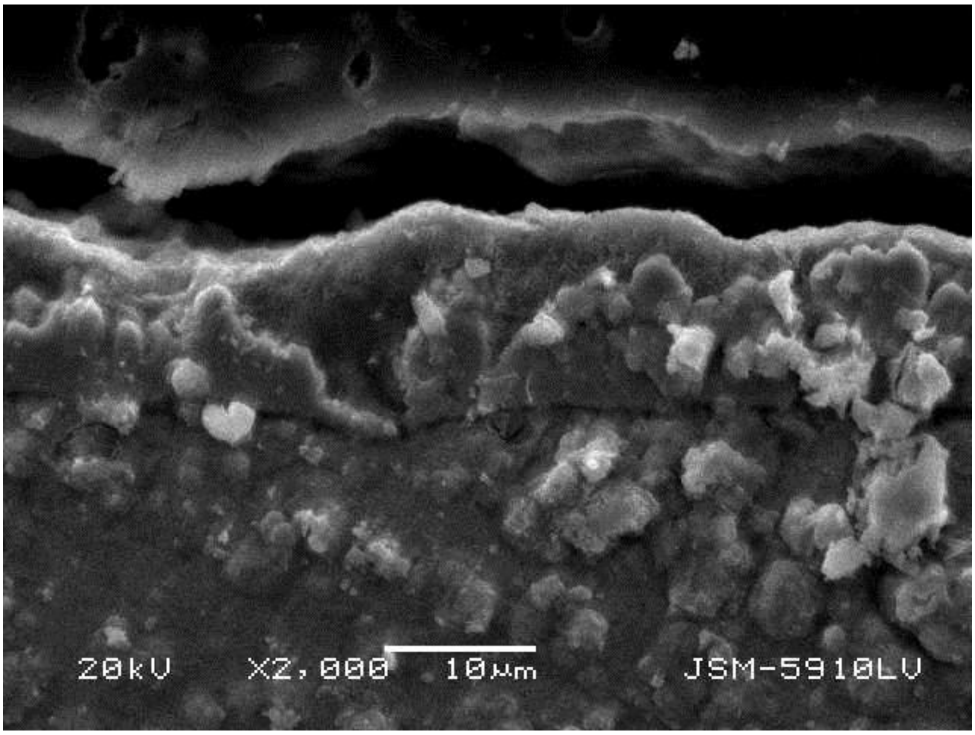

Figure 3.29. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

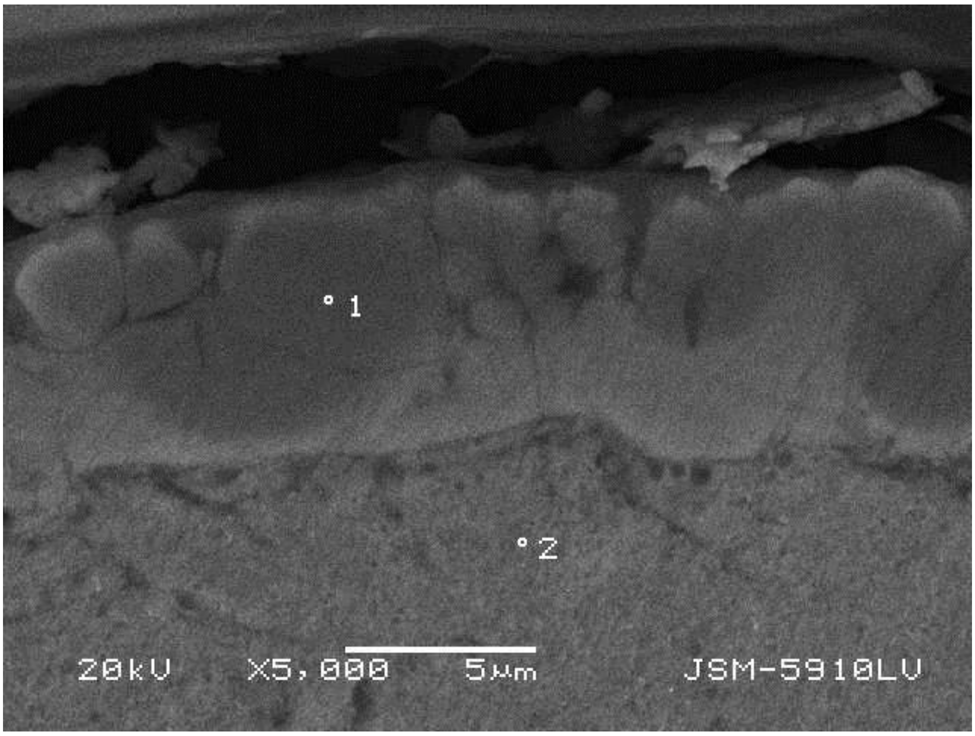

Figure 3.30. SEM - EDS cross section image of alkaline zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (5000x magnification).

EDS analysis in Figure 3.30 marked as point 1 and 2 were explained in Table 3.4 and Table 3.5 and were shown in Figure 3.31 and Figure 3.32. Analyzed sample were exposed 240 h ASTM B 117 neutral salt spray test.

Table 3.4. EDS analysis in Figure 3.30 (point 1).

Spectrum	Fe	Ni	Zn	Total
Spectrum 4	1.08	11.36	87.56	100.00
Max.	1.08	11.36	87.56	
Min.	1.08	11.36	87.56	

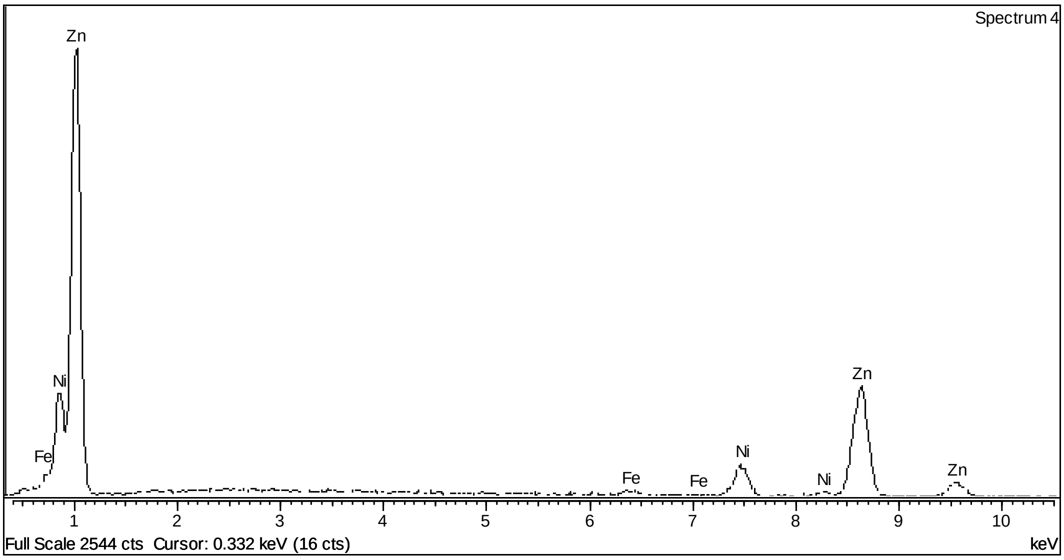

Figure 3.31. EDS analysis of point 1 in Figure 30.

Table 3.5. EDS analysis in Figure 3.30 (point 2).

Spectrum	O	Al	Zn	Total
Spectrum 1	3.57	2.52	93.91	100.00
Max.	3.57	2.52	93.91	
Min.	3.57	2.52	93.91	

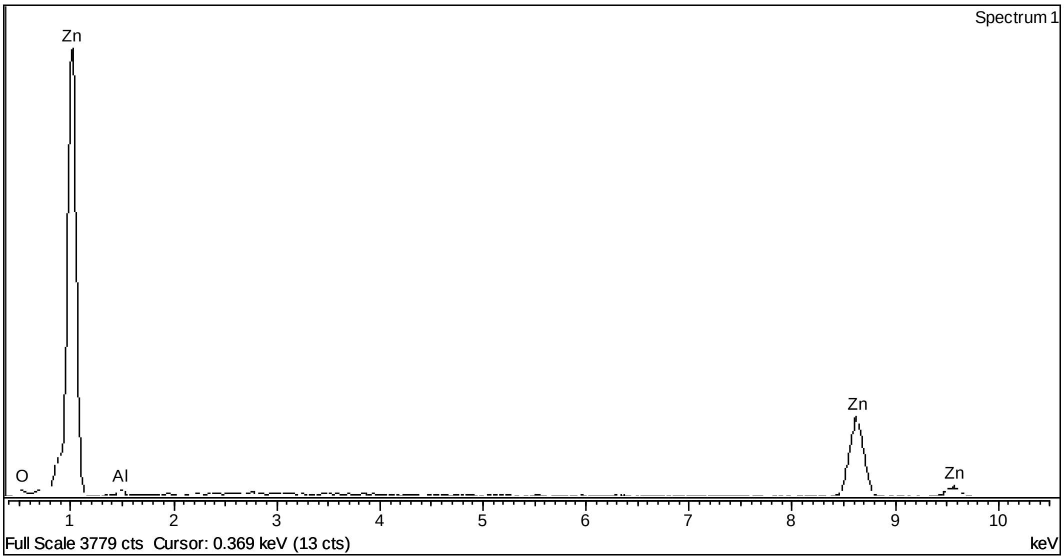

Figure 3.32. All values in Table 3.5 are as weight percent.

Point 1 is middle of the coating and showed 1,08 % Fe, 11,36 % Ni and 87,56 % Zn which indicated no corrosion. Point 2 is middle of the zamak substrate analyzed as % 3,57 O, % 2,52 Al and % 93,91 Zn. This element values show no explosion to corrosion.

Optical microscope and SEM results of alkaline zinc nickel coating on the zamak parts before salt spray test showed smooth and permanent coating. After 240 h salt spray test, black spot areas were detected at optical microscope images and cracks and cleavages were seen at SEM images. 480 h salt spray test showed that larger black spots areas and larger distance between cracks and cleavages were formed, respectively at optical microscope and SEM images. In addition, ZnO was detected as larger salt region at SEM images. It is known that corrosion damages to coating and struggle to find a way to reach base metal.

3.2.3. Acid Zinc Nickel Coating on Casting Parts

Acidic zinc nickel coated casting parts were investigated by optical microscope and scanning electron microscope after 240 h and 480 h neutral salt spray test and also coating without neutral salt spray test.

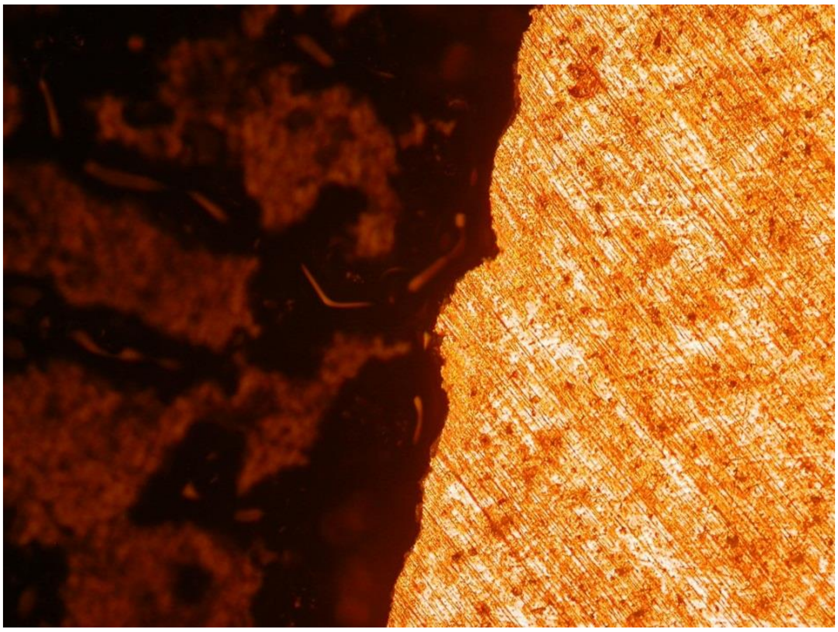

Figure 3.33. Optical microscope cross section image of acid zinc nickel coating on the casting part (1000x magnification).

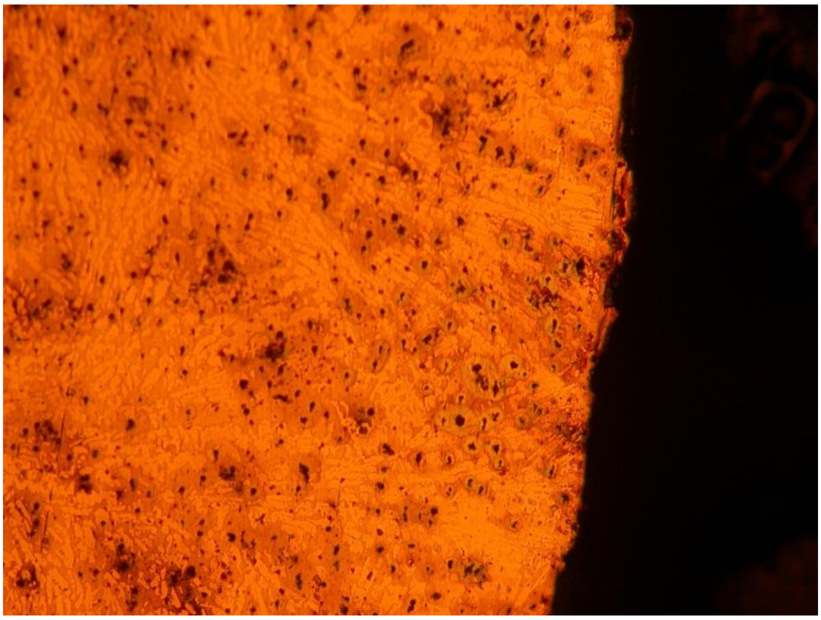

Figure 3.34. Optical microscope cross section image of acid zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (1000x magnification).

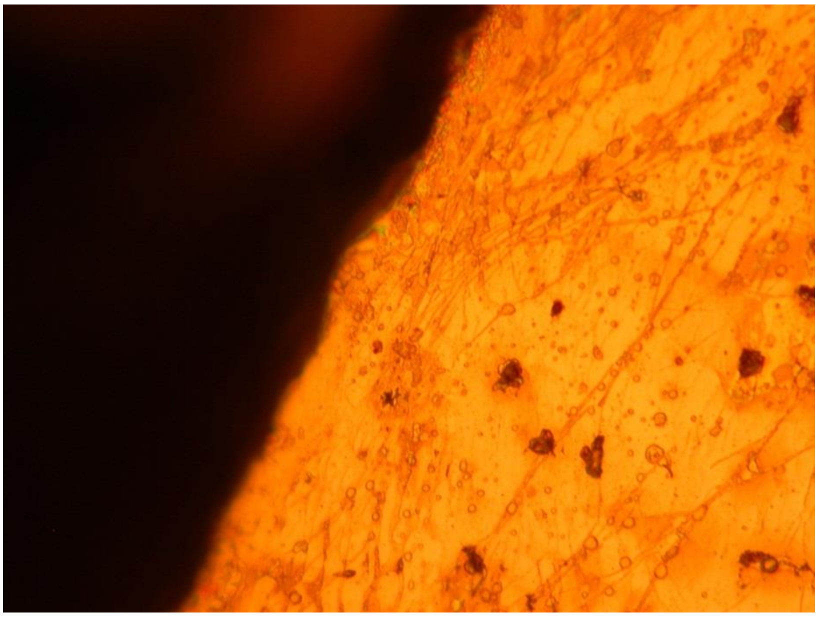

Figure 3.35. Optical microscope cross section image of acid zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (5000x magnification).

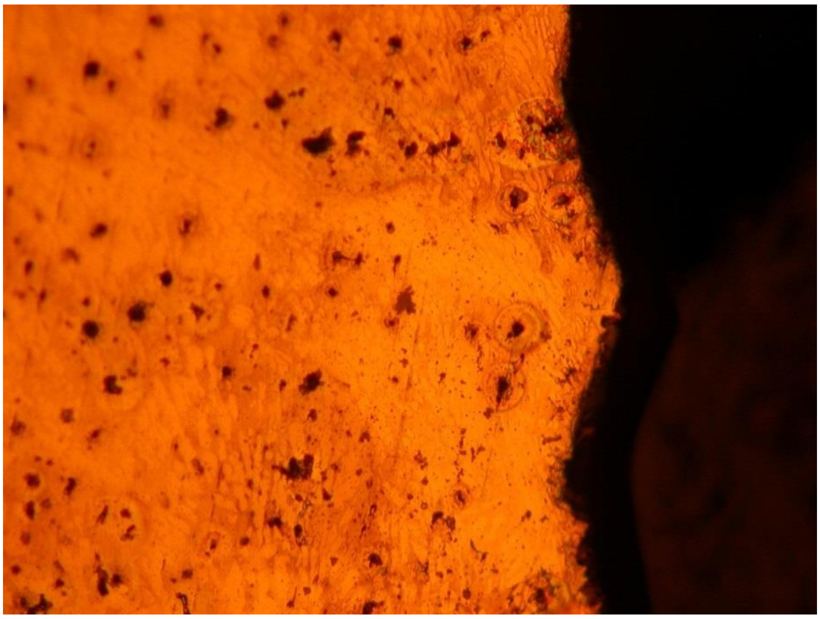

Figure 3.36. Optical microscope cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

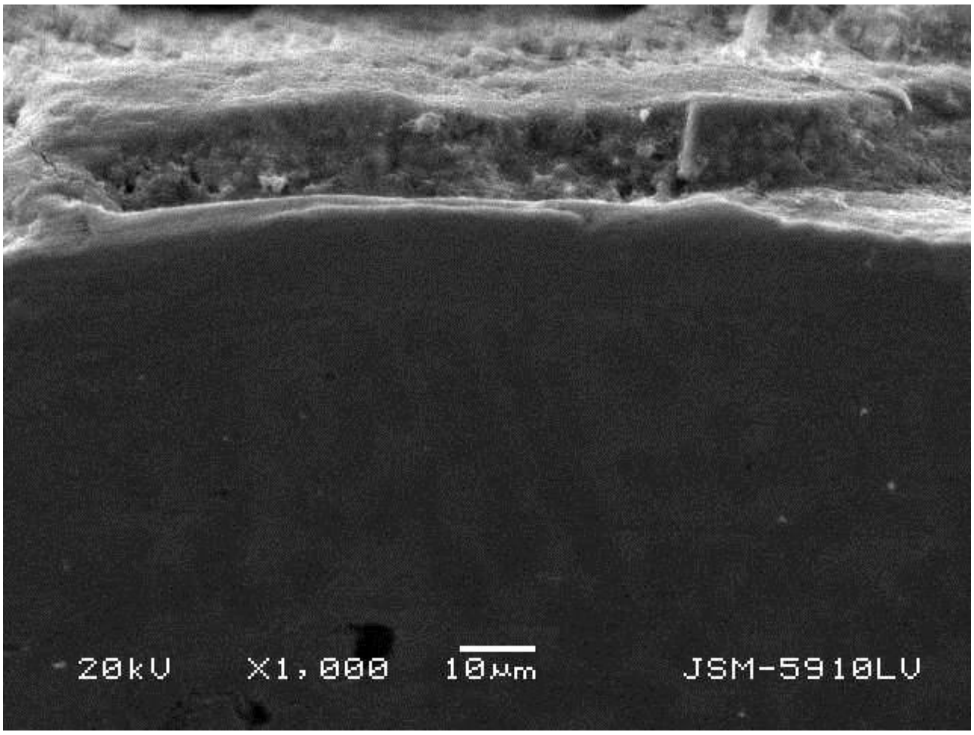

Figure 3.37. SEM cross section image of acid zinc nickel coating on the casting part (1000x magnification).

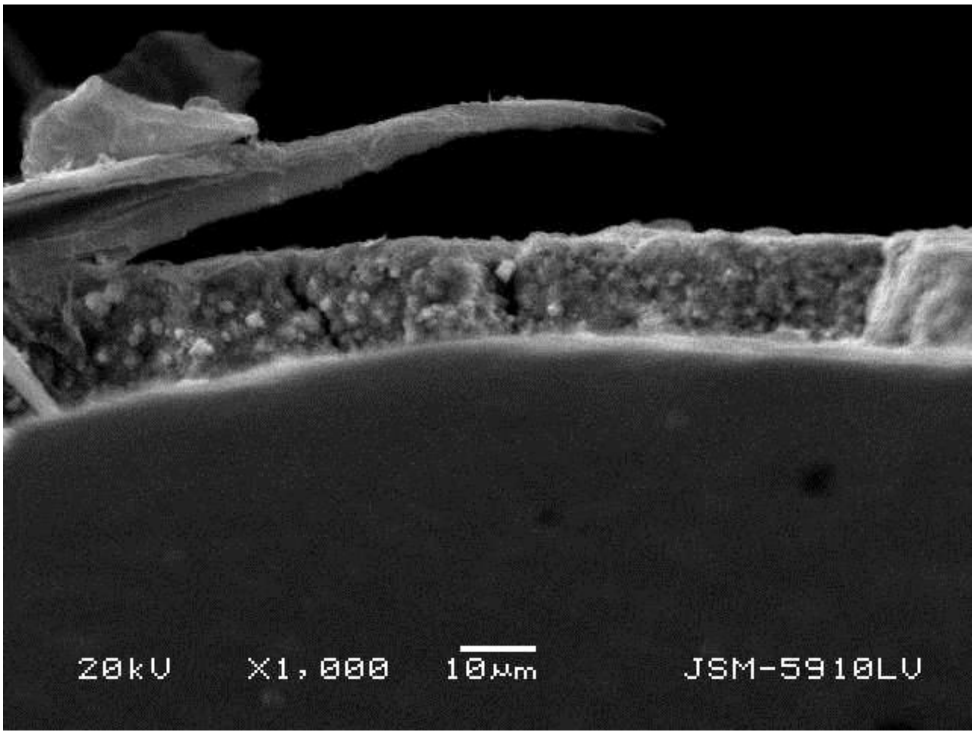

Figure 3.38. SEM cross section image of acid zinc nickel coating on the casting part (1000x magnification).

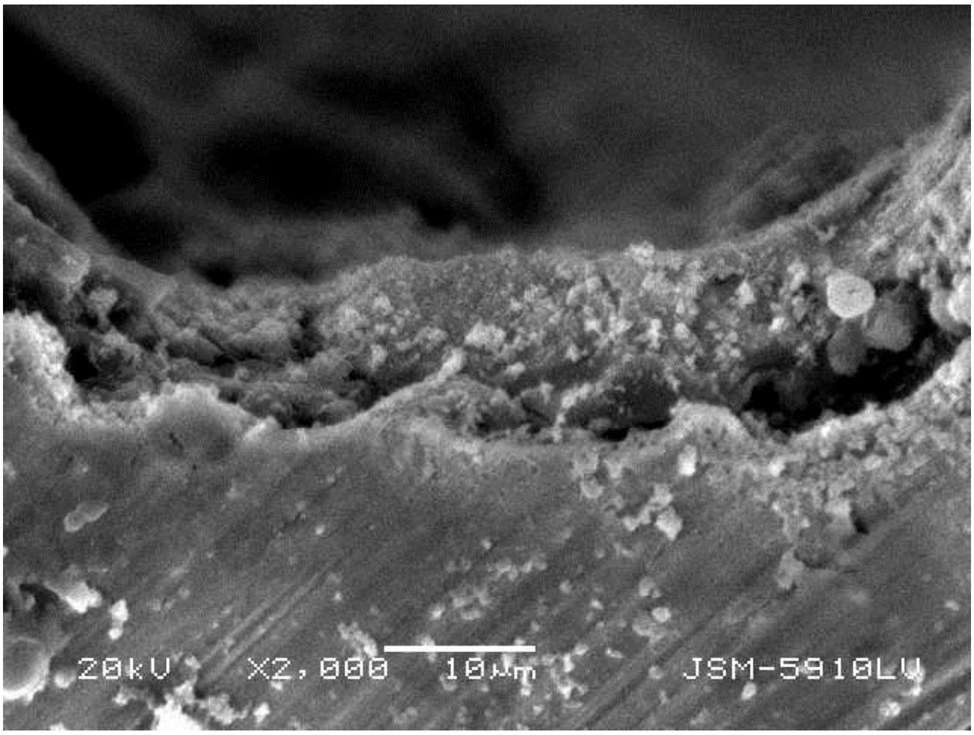

Figure 3.39. SEM cross section image of acid zinc nickel coating on the casting part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

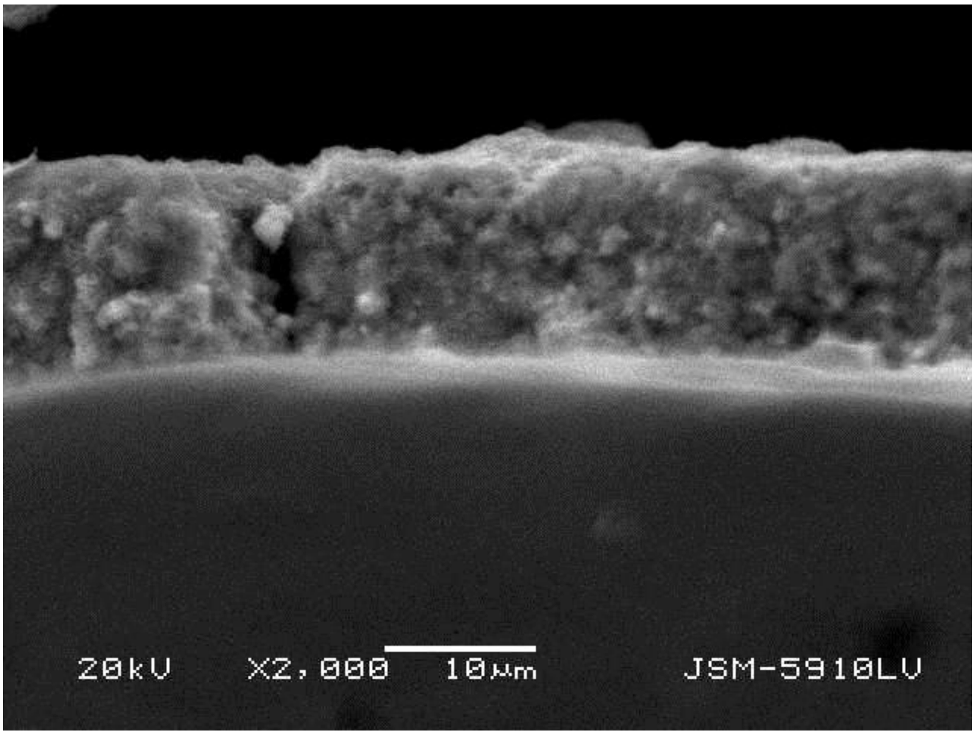

Figure 3.40. SEM cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

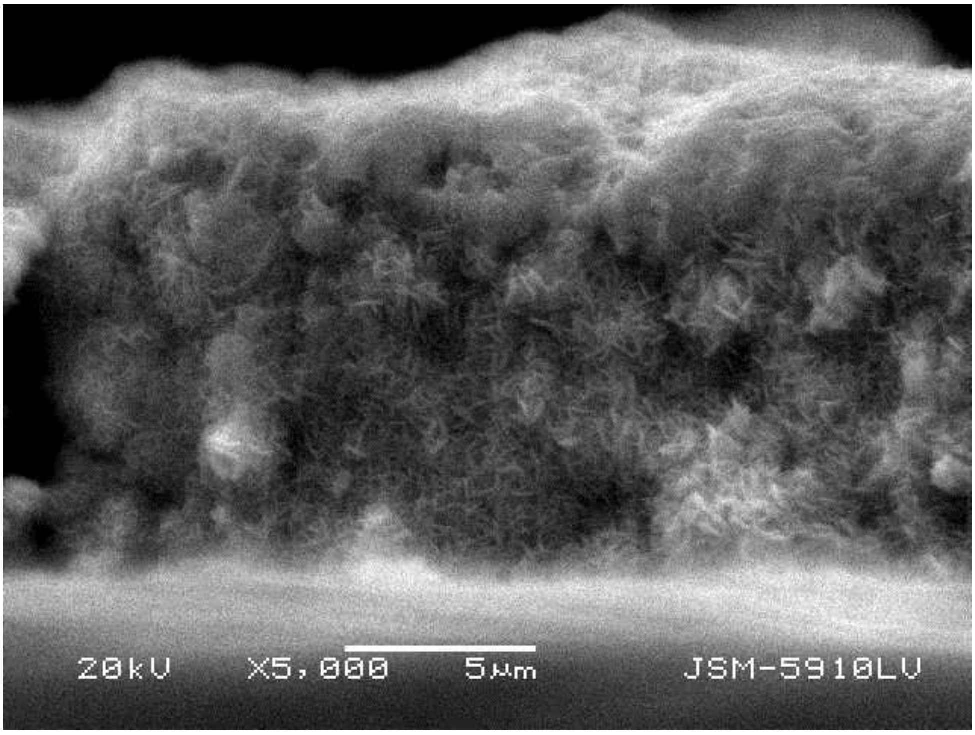

Figure 3.41. SEM cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (5000x magnification).

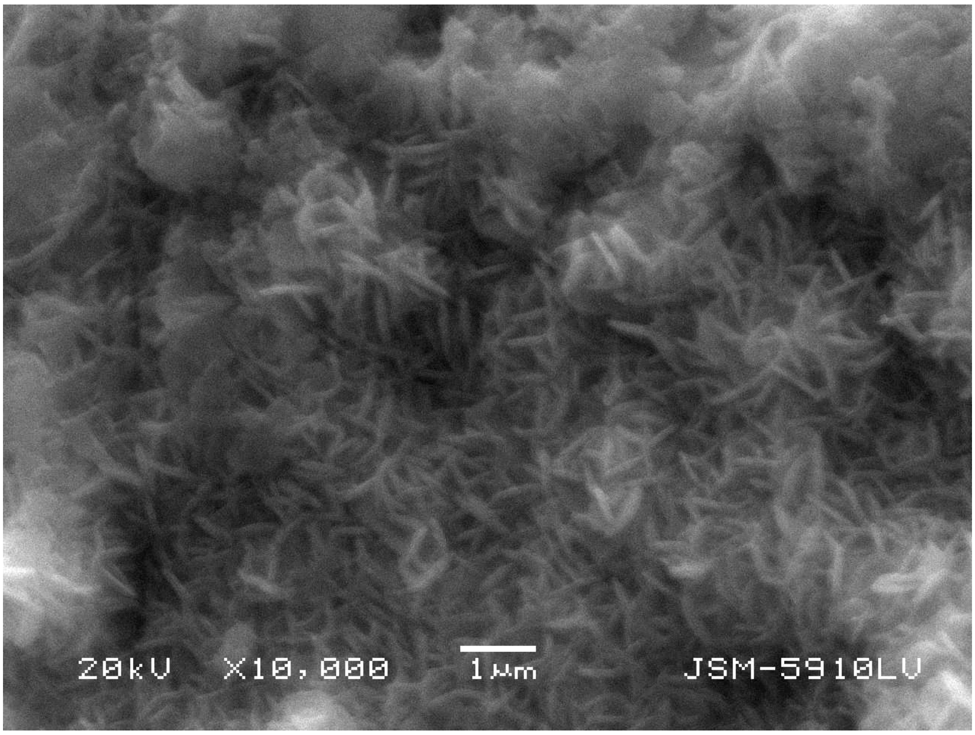

Figure 3.42. SEM cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (10.000x magnification).

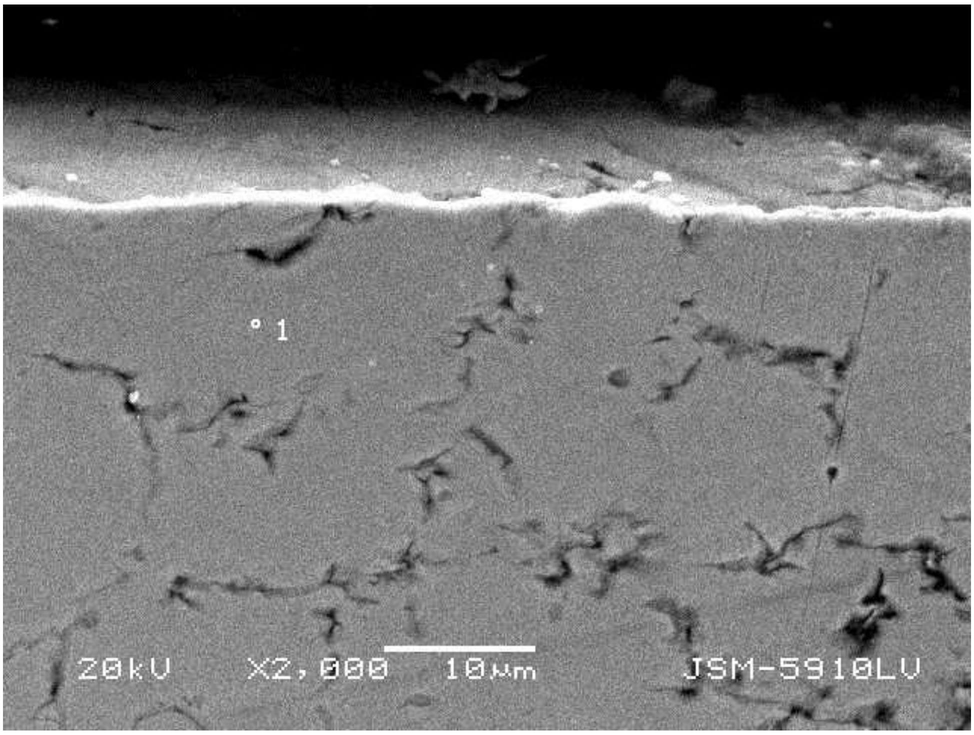

Figure 3.43. SEM - EDS cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

Elements of number 1 as marked on Figure 3.43 was explained on the Table 3.6 and Table 3.7 and showed as graph on Figure 3.44 and Figure 3.45.

Table 3.6. All elements on Figure 3.44 and Figure 3.45 analyzed for point 1 on Figure 3.43.

Spectrum	Si	Mn	Fe	Total
Spectrum 1	3.21	0.32	96.47	100.00
Max.	3.21	0.32	96.47	
Min.	3.21	0.32	96.47	

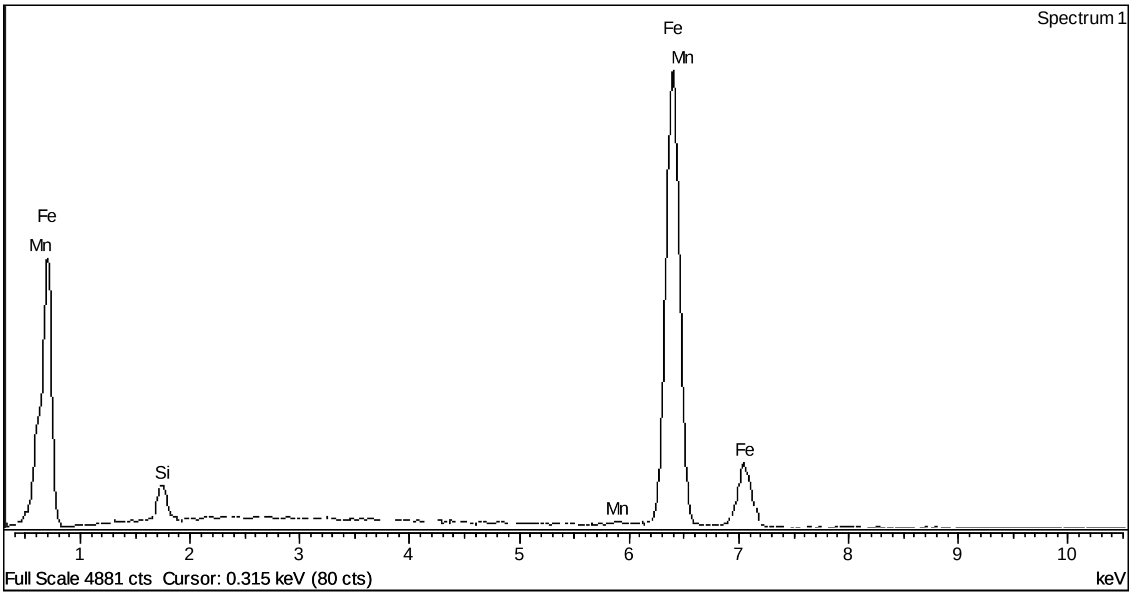

Figure 3.44. All values in Table 3.6 are as weight percent.

Table 3.7. All elements analyzed for point 2 on Figure 3.43.

Spectrum	O	Zn	Total
Spectrum 1	25.08	74.92	100.00
Max.	25.08	74.92	
Min.	25.08	74.92	

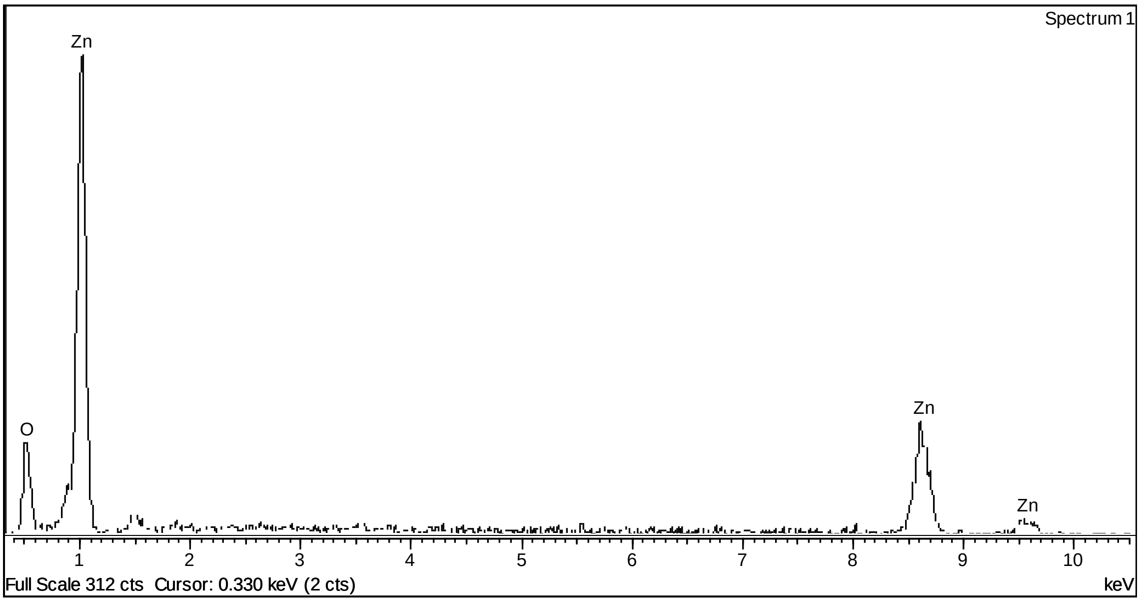

Figure 3.45. All values in Table 3.7 are as weight percent.

Point 1 and point 2 on Figure 3.43 by EDS. Analyzed sample exposed 480 h ASTM B 117 neutral salt spray test. Point 2 is corner of the coating analyzed as 25,08% O, 74,92% Zn. This element values show to expose corrosion. Point 1 is middle of the casting substrate analyzed as 3,21% Si, 0,32% Mn and 96,47% Fe. This element values show not to expose corrosion.

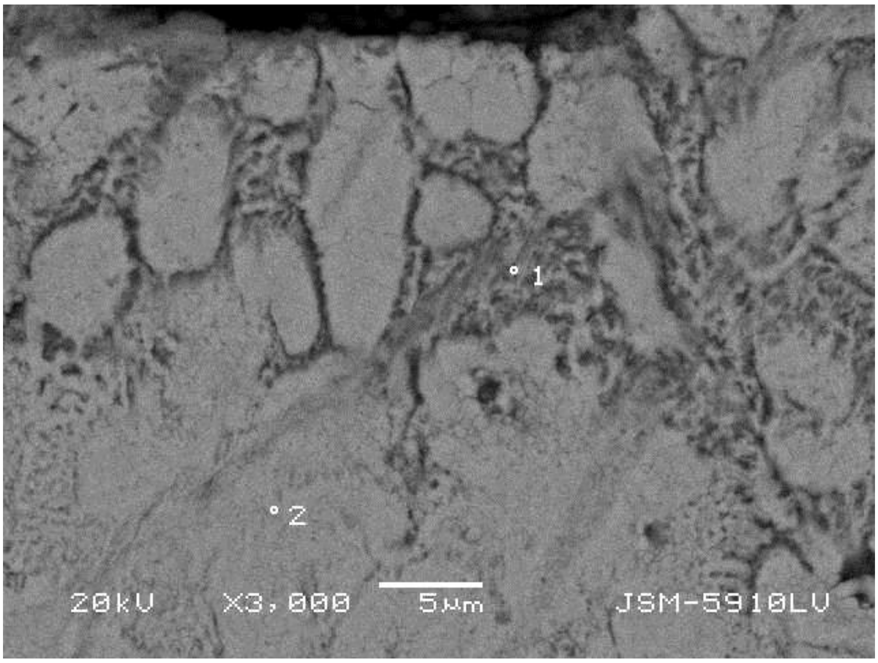

Figure 3.46. SEM – EDS cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (3000x magnification).

Elements of number 2 as marked on Figure 3.46 was explained on the Table 3.8 showed as graph on Figure 3.47.

Table 3.8. All elements on Figure 3.46 analyzed for point 1 on Figure 3.46.

Spectrum	O	Cl	Zn	Total
Spectrum 1	26.36	13.92	59.72	100.00
Max.	26.36	13.92	59.72	
Min.	26.36	13.92	59.72	

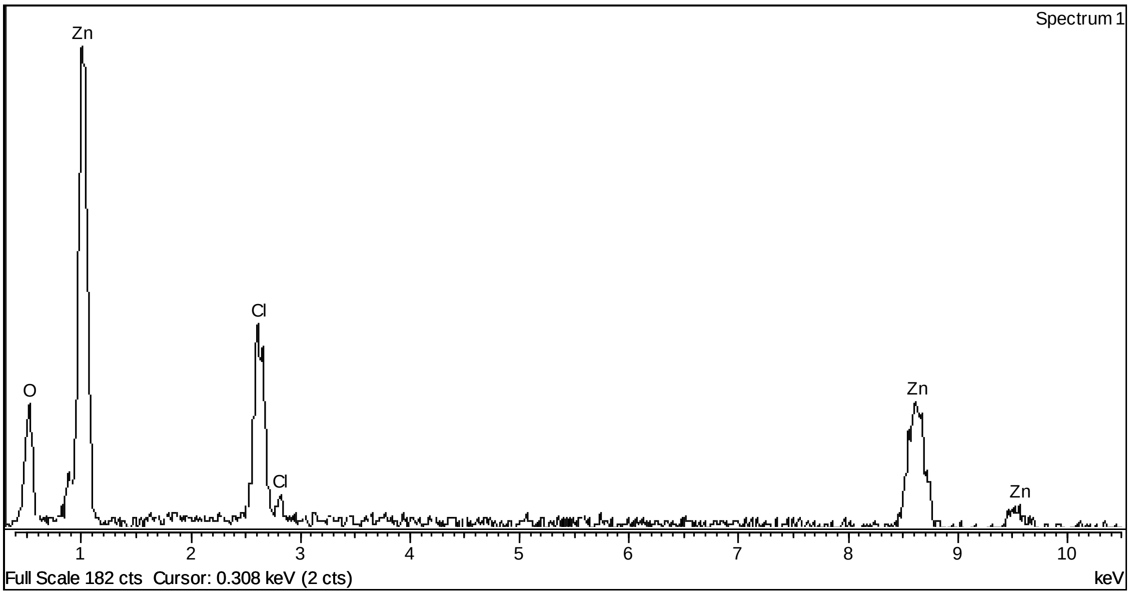

Figure 3.47. All valuess in Table 3.8 are as weight percent.

Point 1 and point 2 on Figure 3.81 and Figure 3.46 by EDS. Analyzed sample exposed 480 h ASTM B 117 neutral salt spray test. Point 1 is corner of the coating analyzed as 26,36% O, 13,92% Cl and 59,72% Zn. This element values show to expose corrosion.

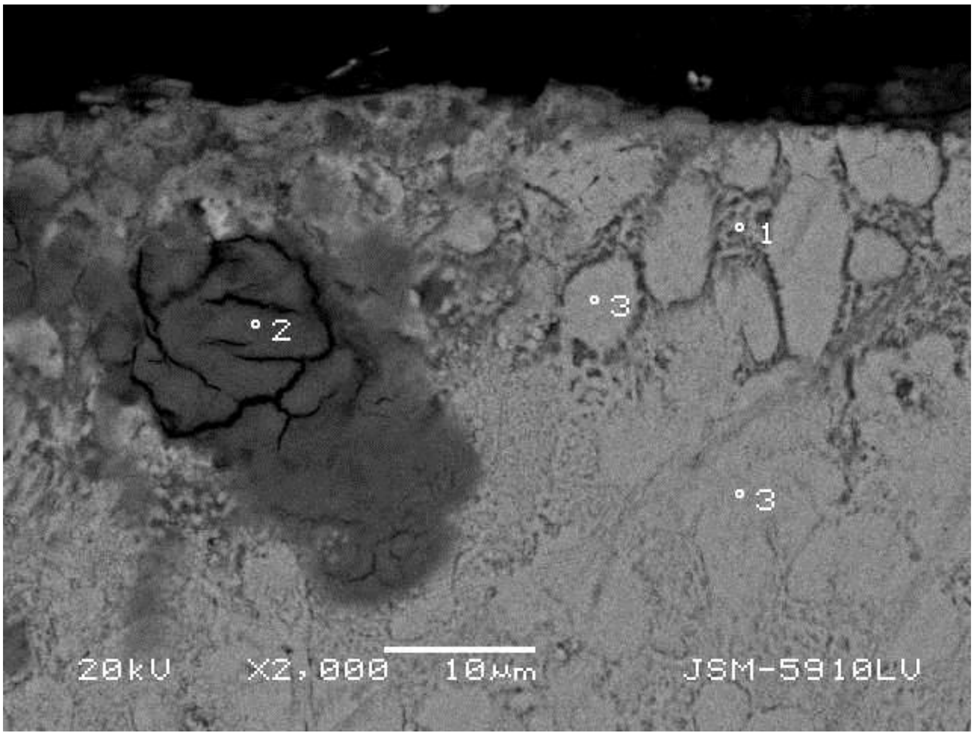

Figure 3.48. SEM – EDS cross section image of acid zinc nickel coating on the casting part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

Point 3 as marked on Figure 3.48 was explained on the Table 3.9 showed as graph on Figure 3.49.

Table 3.9. All elements on Figure 3.48 analyzed for point 3 on Figure 3.48.

Spectrum	Zn	Total
Spectrum 1	100.00	100.00
Max.	100.00	
Min.	100.00	

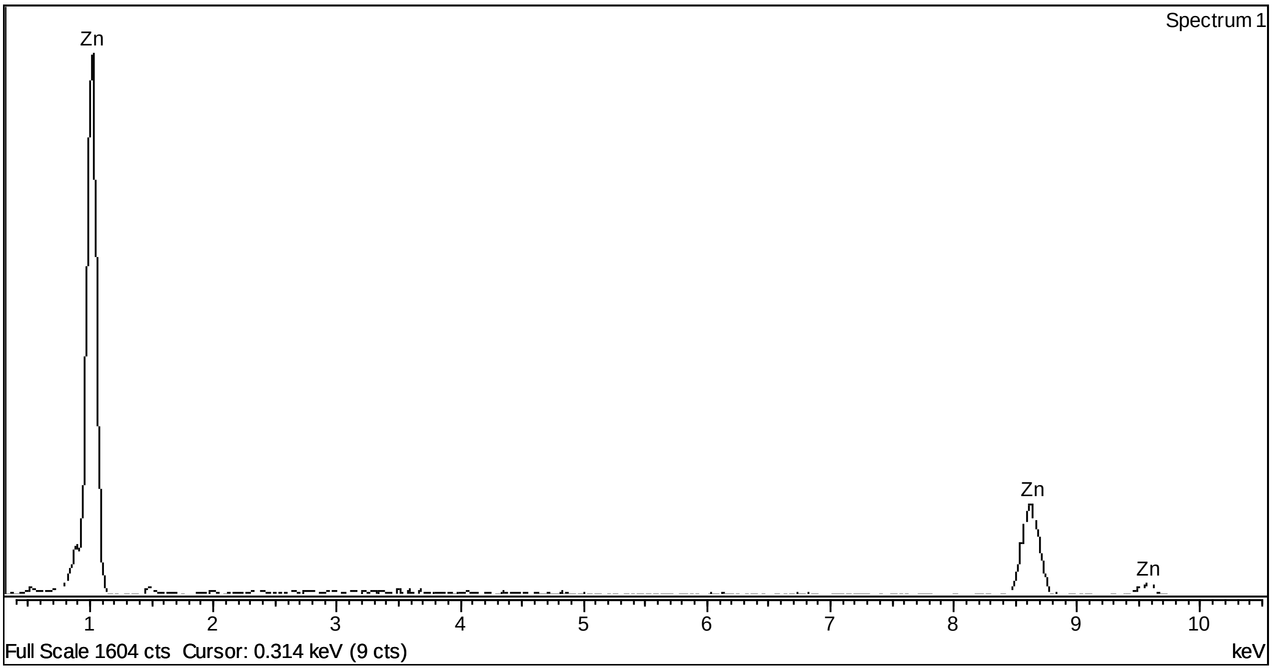

Figure 3.49. All values in Table 3.9 are as weight percent.

Point 1, point 2 and point 3 were investigated on Figure 3.48 by EDS. Analyzed sample exposed 480 h ASTM B 117 neutral salt spray test. Point 3 is middle of the coating analyzed as 100% Zn. This element values could be wrong measurement or zinc collected on the point.

Optical microscope and SEM results of acid zinc nickel coating on the zamak parts before salt spray test showed smooth and permanent coating. After 240 h salt spray test, black spot areas were detected at optical microscope images and cracks and cleavages were seen at SEM images. After 480 h salt spray test, bigger black spots areas were detected at optical microscope images and cracks and cleavages were seen at SEM images. In addition, ZnO is seen as larger salt regions at SEM images. Corrosion damages to coating and struggle to find a way to reach base metal. EDS data supports our comment. It's important that acid zinc nickel coating bath is more aggressive than alkaline zinc nickel coating bath. Therefore acid zinc coating bath damages to surface of substrate part before starting to coat it.

3.2.4. Acidic Zinc Nickel Coating on Zamak Parts

Acidic zinc nickel coated zamak parts were investigated by optical microscope and scanning electron microscope after 240 h and 480 h neutral salt spray test and also coating without neutral salt spray test.

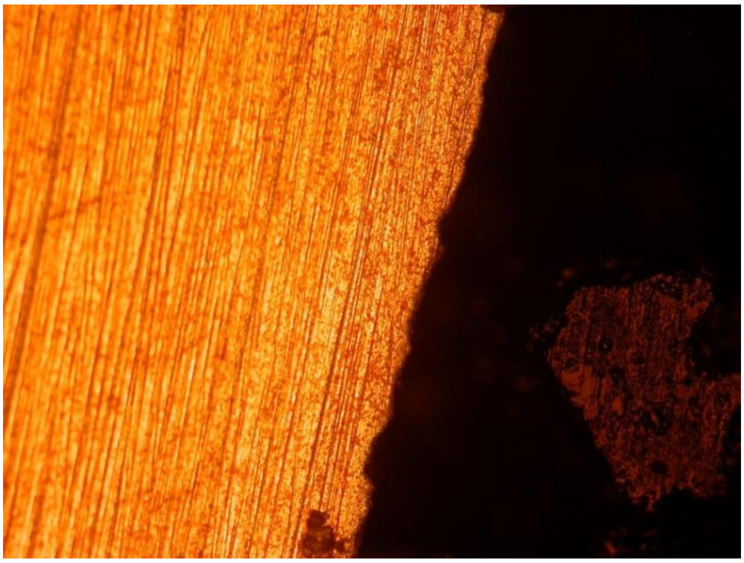

Figure 3.50. Optical microscope cross section image of acid zinc nickel coating on the zamak part(1000x magnification).

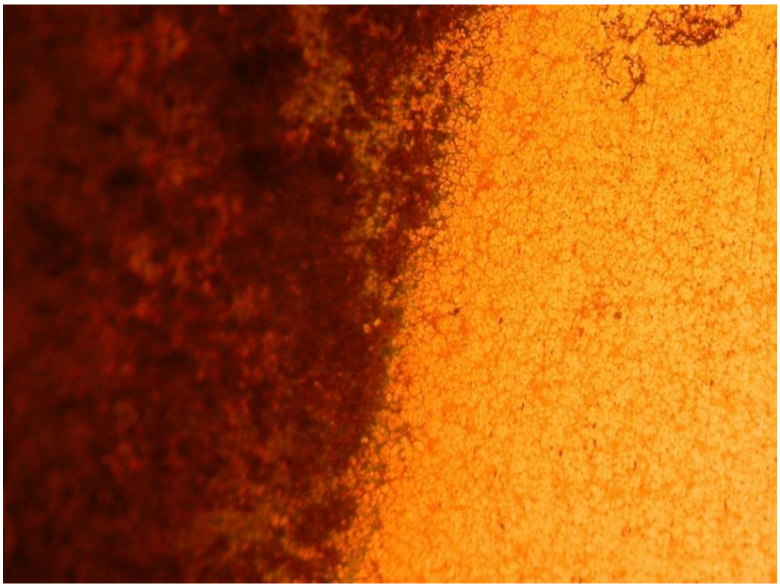

Figure 3.51. Optical microscope cross section image of acid zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (x magnification).

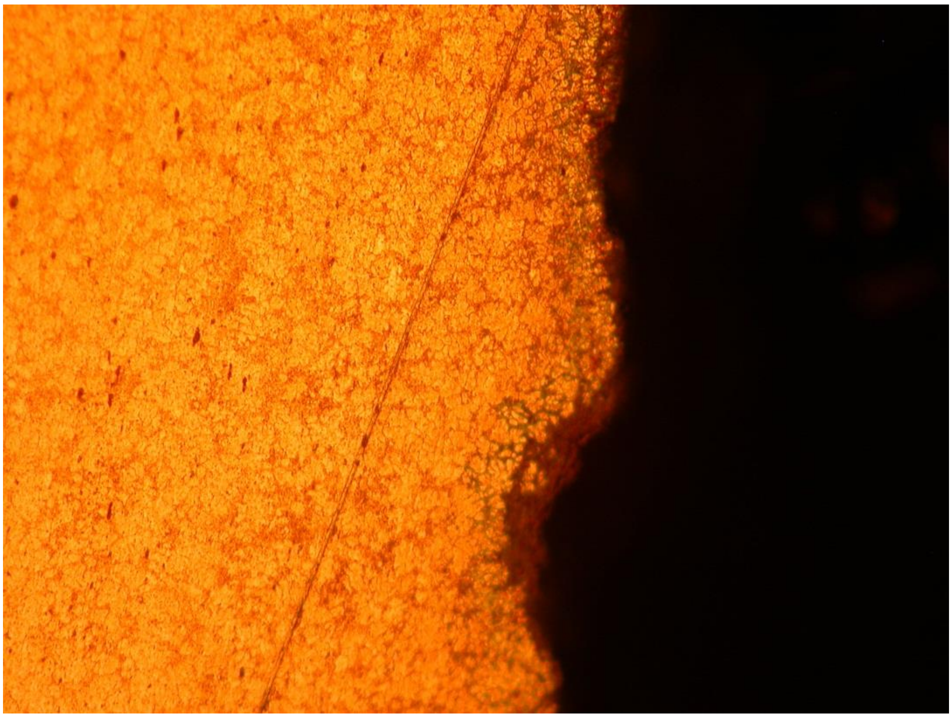

Figure 3.52. Optical microscope cross section image of acid zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

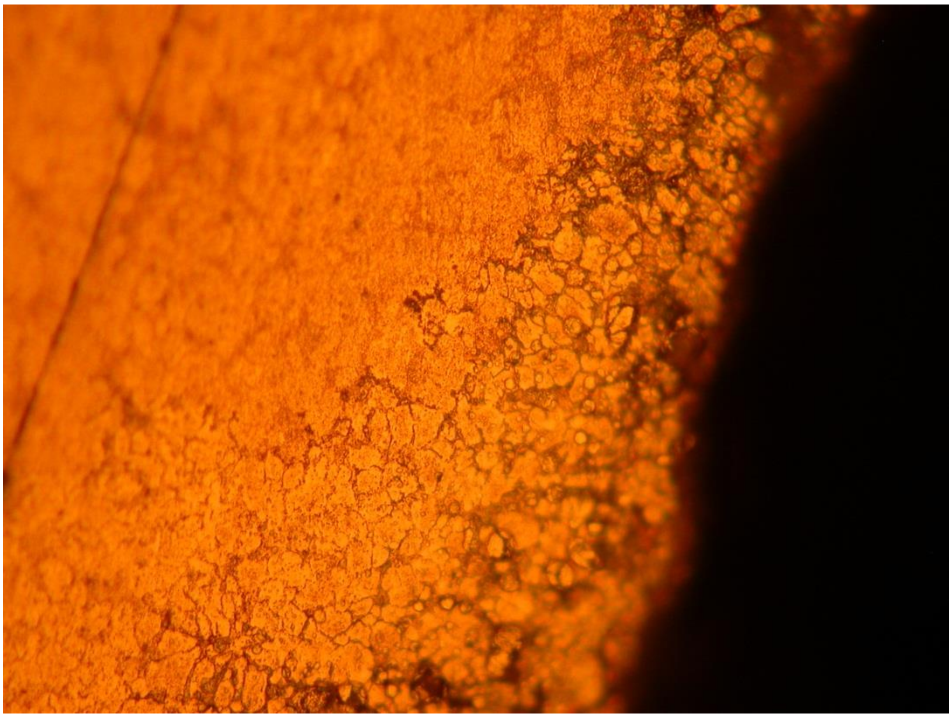

Figure 3.53. Optical microscope cross section image of acid zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (5000x magnification).

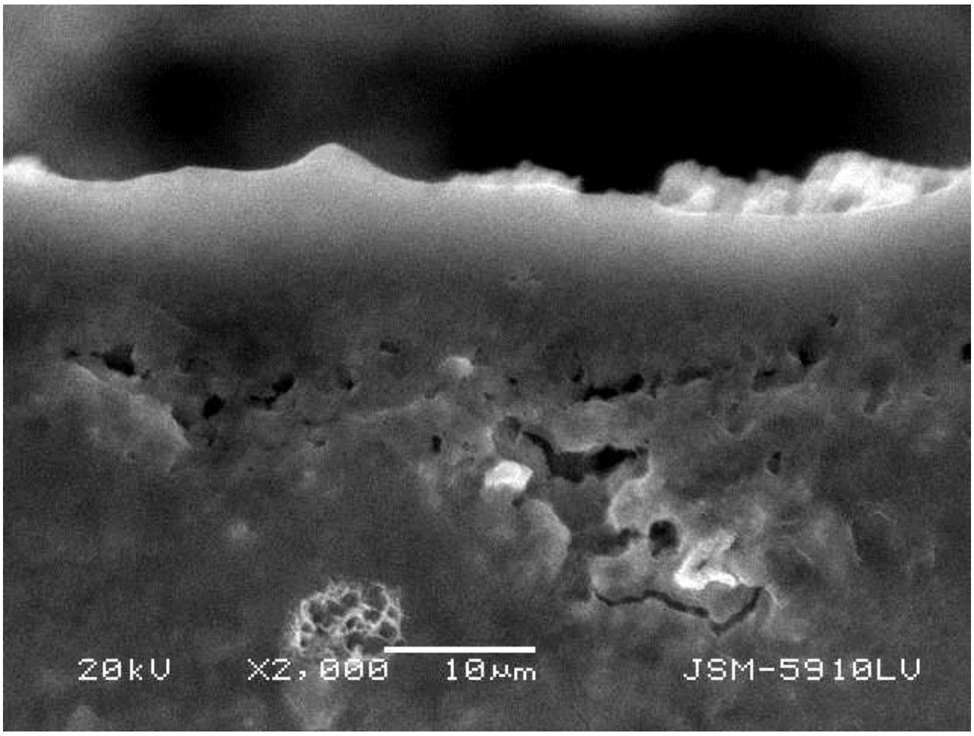

Figure 3.54. SEM cross section image of acid zinc nickel coating on the zamak part (2000x magnification).

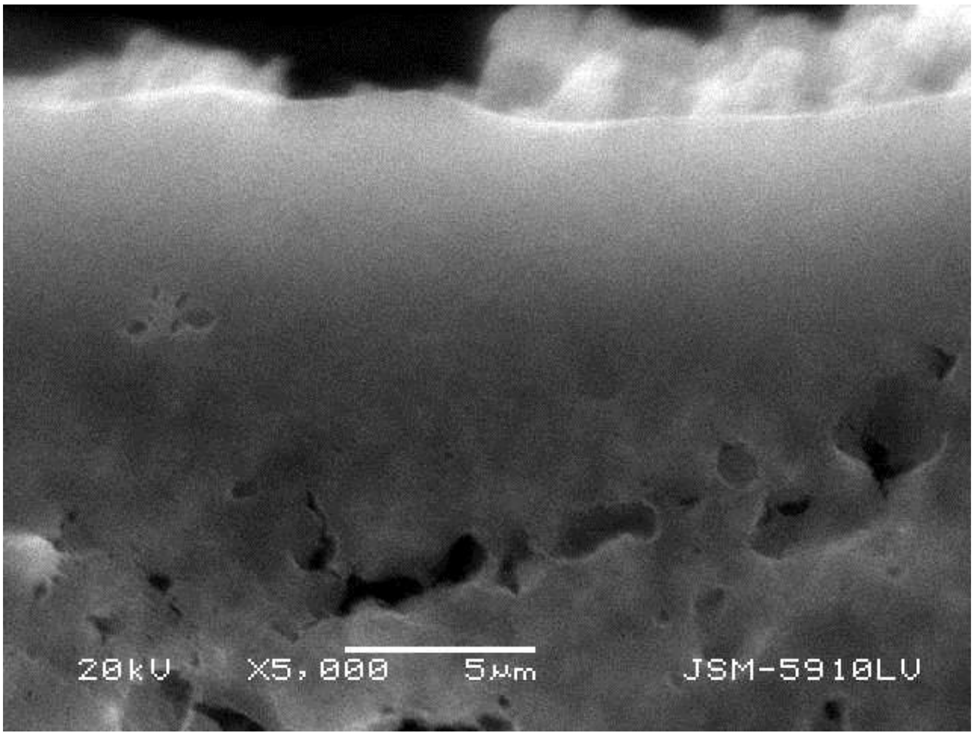

Figure 3.55. SEM cross section image of acid zinc nickel coating on the zamak part (5000x magnification).

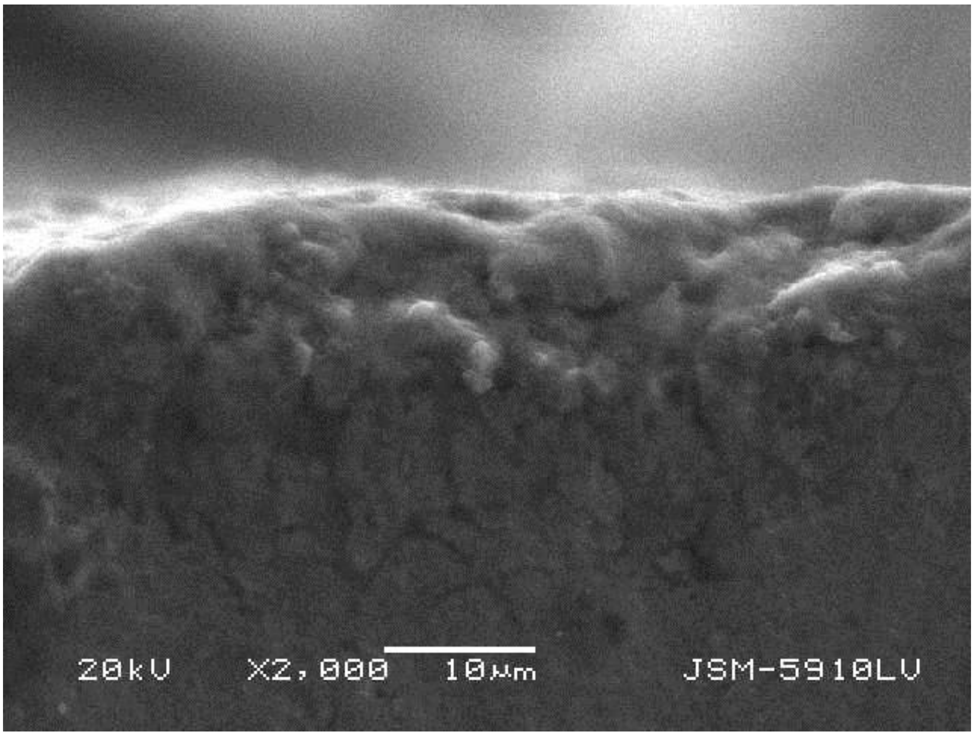

Figure 3.56. SEM cross section image of acid zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (2000x magnification).

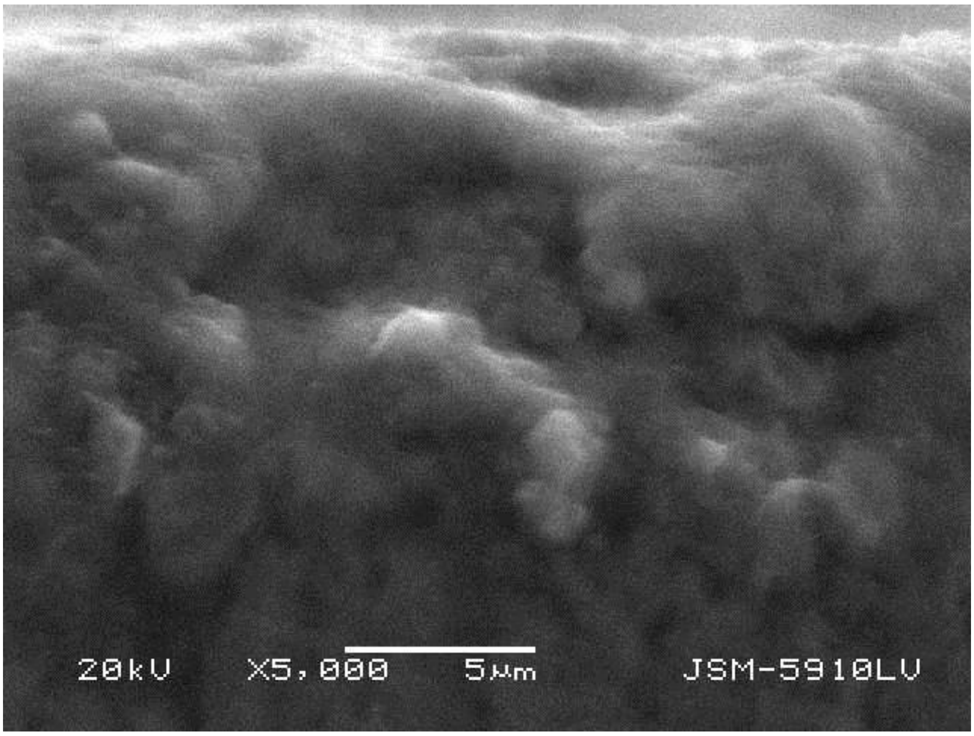

Figure 3.57. SEM cross section image of acid zinc nickel coating on the zamak part after 240 h ASTM B 117 neutral salt spray test (5000x magnification).

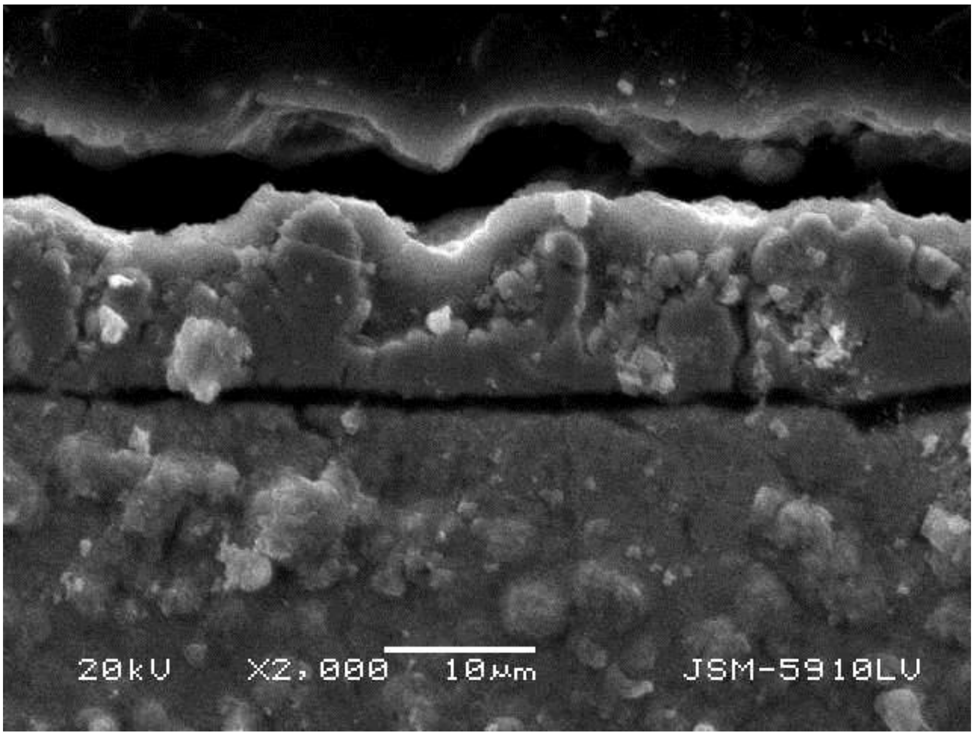

Figure 3.58. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

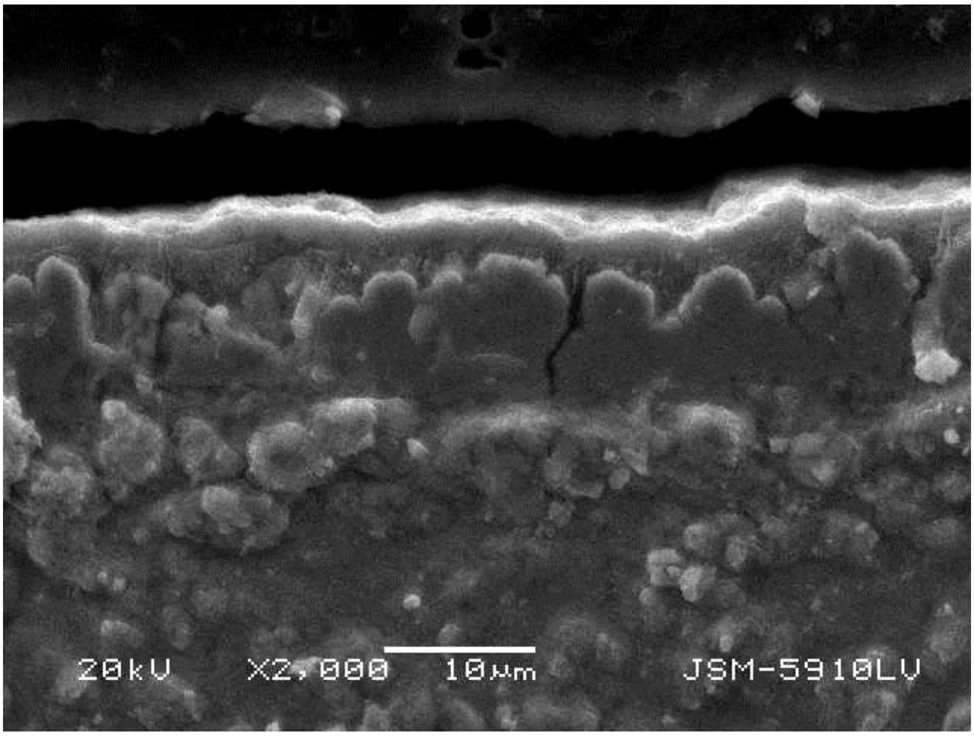

Figure 3.59. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

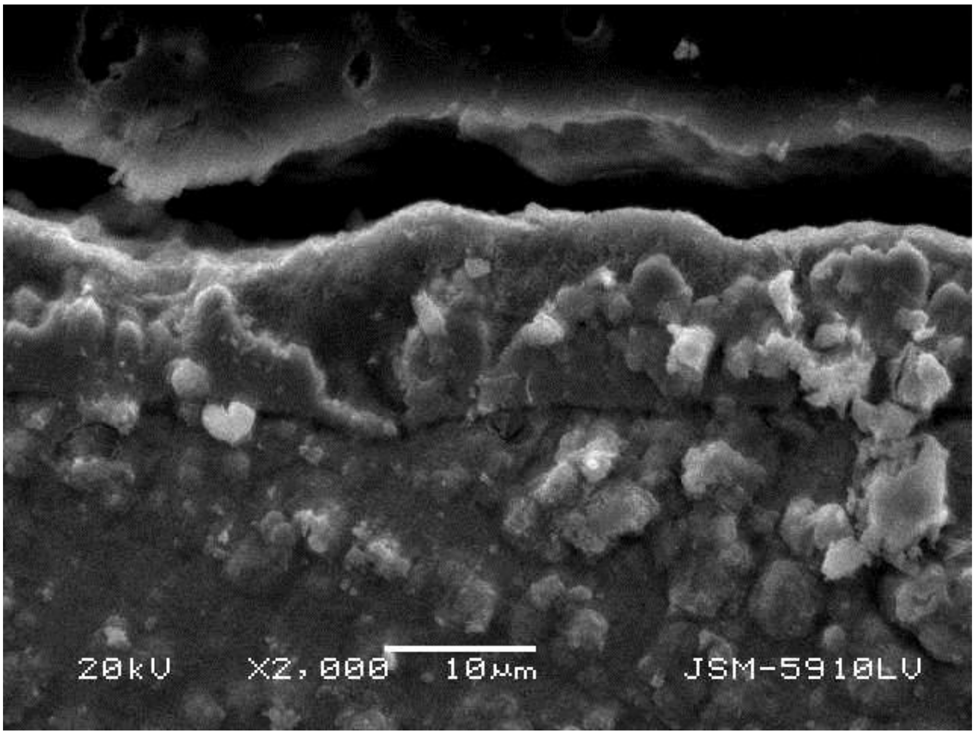

Figure 3.60. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (2000x magnification).

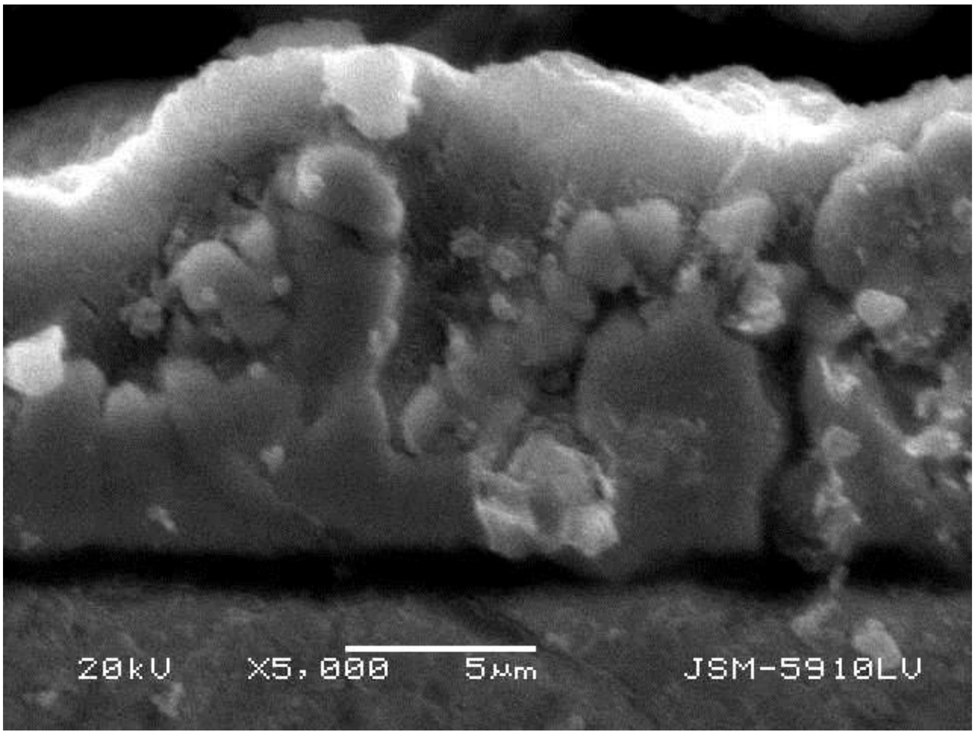

Figure 3.61. SEM cross section image of alkaline zinc nickel coating on the zamak part after 480 h ASTM B 117 neutral salt spray test (5000x magnification).

Optical microscope and SEM results of acid zinc nickel coating on the zamak parts before salt spray test showed smooth and permanent coating. After 240 h salt spray test, black spot areas were detected at optical microscope images and cracks and cleavages were seen at SEM images. 480 h salt spray test showed that larger black spots areas and larger distance between cracks and cleavages were formed, respectively at optical microscope and SEM images. After 480 h salt spray test, bigger black spots areas are seen clearly at optical microscope images and also the distance between cracks and cleavages are seen to increase at SEM images. In addition, ZnO is seen as larger salt reagions at SEM images. Corrosion damages coating and struggle to find a way to reach base metal. EDS data supports our comment. It's important that acid zinc nickel coating bath is more aggressive than alkaline zinc nickel coating bath. Therefore acid zinc coating bath damages to surface of substrate part before starting to coat it. Moreover acid zinc coating bath damages zamak parts more than casting parts. Because zamak consist of high zinc and aluminum.

3.3. XRD Values of Zamak and Zinc Nickel Coating

Zamak substrate and zinc nickel coating was examined by XRD (Figure 3.62). Figure 3.62a shows zinc element that is zamak substrate. Figure 3.62b shows zinc and nickel that is zinc nickel coating layer.

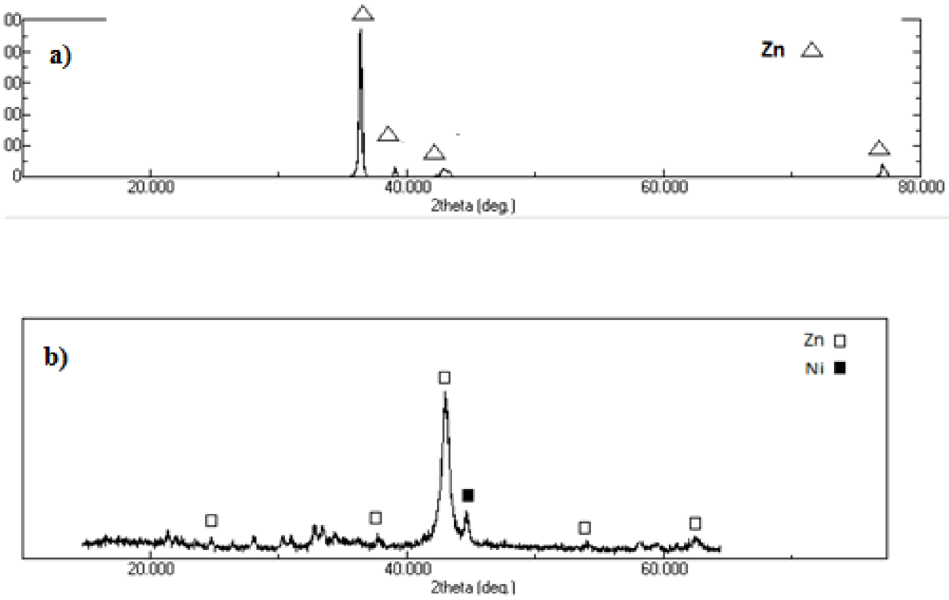

Figure 3.62. XRD pattern of a) zamak and b) Zn-Ni coating.

4. COCLUSIONS

The result of literature and experimental investigations prove that acid and alkaline zinc nickel alloy deposits have similar surface morphologies, texture and gamma phase formation. Therefore, acid zinc nickel electrolyte can be used on all substrate materials that can be coated by alkaline zinc nickel. But the investigations show that acid zinc nickel coating bath is more aggressive than alkaline zinc nickel coating bath. Therefore, acid zinc coating bath damages substrate surface before coating. However, after damaging the surface, the coating is much faster than alkaline bath. As a result, acidic coatings have similar corrosion resistance with alkaline coatings due to increased coating thickness and better adhesion. In addition, complex shaped casting parts which have high carbon content can't be coated by alkaline zinc nickel electrolyte due to low current efficiency. But these parts can be easily coated by acid zinc nickel electrolyte. In the automotive industry, brake calipers can be easily coated with zinc nickel alloy deposit by acid zinc nickel electrolyte. As a result high corrosion resistance is achieved on the casting parts. The investigations explained that acidic and alkaline zinc nickel alloy deposits had similar properties not only on the casting parts but also zamak parts.

Increasing demands on corrosion protection in the field of sacrificial coatings require more and more the use of zinc alloys. Among these, zinc nickel has proven to provide the best performance for commercially available zinc alloys. Research has shown that it is crucial to deposit this coating with the Ni_2Zn_{11} gamma phase structure to achieve this benefit in corrosion protection. After difficult first trials with acidic based zinc nickel electrolytes quickly the alkaline zinc nickel solutions have gained acceptance in the researches by providing more stable and reliable results. Nevertheless, the development of a new acidic zinc nickel electrolytes shows that it is possible now also to achieve the same deposit properties of deposits as alkaline zinc nickel deposits. With the successful introduction to the brake caliper industry and the first promising results from coating fasteners one can say, that a new chapter in zinc nickel plating has started.

The results show that the acidic and alkaline zinc nickel electrolytes produced zinc nickel alloy coatings on casting and zamak parts. Both coatings had identical surface morphology, texture and gamma phase formation. The examined deposits indicated good performance in terms of nickel incorporation to produce relevant current density spectrum.

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