

Original research article

The influence of surface imperfections on phosphate coating performance of nodular cast iron substrates

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Abstract: The service performance of the nodular cast iron wheel hub was modelled by investigating the effect of surface morphology and characteristics on the phosphate coating size, morphology, and corrosion resistance properties of the zinc phosphate conversion coated cast iron substrates. The machined and unmachined surfaces as well as the coatings were examined by a scanning electron microscope coupled with an energy-dispersive X-ray spectrometer (SEM-EDX). The influence of the substrates' surface imperfections on the phosphate coating and the subsequent corrosion resistance was assessed and rated according to the standard procedures. Surface analysis of the machined and unmachined cast iron hubs reveals the existence of graphite and foreign material inclusions on the substrate surface that impact the phosphate coating properties and resistance to corrosion. The average phosphate coating crystal size is 1.74 μm and 2.58 μm for the unmachined and machined cast iron substrates, respectively. The corrosion resistance of the coated unmachined wheel hub surfaces was rated poor and disapproved based on the application requirements. The poor corrosion resistance was ascribed to the influence of the substrates' surface characteristics on the coating adhesion to the substrate. However, the cast surfaces should be properly shot-blasted to remove any adhere foreign materials on the as-cast skin to enhance the coating adhesion.

Keywords: spheroidal cast iron; material characterization; phosphate coating; wheel hub assembly; general corrosion; microstructure; SEM-EDS; accelerate corrosion salt spray

Received: 29 May 2023; Accepted: 24 June 2023; Published Online: 21 August 2023

1. Introduction

A crucial element of the steering system is the wheel hub assembly, which is an automotive part used in most vehicles-passenger vehicles, and light and heavy trucks. Because the hub maintains the wheel's connection to the vehicle while allowing unfettered rotation. The wheel hub assembly is in the centre of each wheel, where the wheel bearings are located. Without wheel bearings, the wheels on vehicles wouldn't revolve smoothly which is the major purpose of the wheel hub assembly. The truck wheel hub is the inner shape of the tire, is used to support the barrel body of the tire, and the centre is assembled on the wheel hub shaft. Along with bearings, the hub assembly is also responsible for housing the speed sensors that manage the vehicle's anti-lock braking system. Without a properly functioning hub assembly, the vehicle's anti-lock braking system and traction control would be compromised, making the automobile unsafe to drive^[1]. The bearings, hub, rotor, seal, and drive shaft itself make up most of the vehicle wheel hub assembly. Each of these components must work in unison with the others to manage the pressures at the braking to ensure safe

handling of the vehicle during regular use. A typical wheel hub assembly for a truck is shown in **Figure 1**.



Figure 1. A typical wheel hub (a) and wheel hub assembly (b) for trucks.

The wheel hubs for truck vehicles are usually manufactured from cast iron materials because of their inherent properties and manufacturability. Cast irons are multicomponent ferrous alloys that have found applications in several engineering components^[2–6]. The major constituents of cast iron are iron, carbon, and silicon, and occasionally contain different minor alloying elements. Because of the higher carbon content than that of steel, the structure of cast iron exhibits a richer carbon phase. Cast iron can solidify into either the stable iron-graphite system or the thermodynamically metastable Fe-Fe₃C system, principally depending on composition, cooling rate, and melt treatment. Iron carbide (Fe₃C) is the rich carbon phase in the eutectic when the metastable solidification path is taken; when the stable solidification path is taken, graphite is the rich carbon phase. Based on the graphite morphology, cast irons can be classified as flake (lamellar) graphite, spheroidal (nodular) graphite, compacted (vermicular) graphite, and temper graphite^[7,8]. Among the different classifications of cast irons, spheroidal graphite iron (also known as ductile or nodular graphite cast iron) is typically used to manufacture truck wheel hubs due to its superior mechanical properties as compared to other cast iron materials (**Table 1**). Nodular iron has excellent castability, superior damping capacity, and a good combination of strength, ductility, and toughness^[9]. Over several decades, the automotive industry has used spheroidal graphite cast iron more and more frequently. It is more ductile and has stronger fatigue strength than grey cast irons, and its static strength is comparable to cast steel. Its excellent castability and machinability combined with its medium level of stress resistance make it a viable solution for safety-critical applications and components. Typical applications of nodular cast iron in the automotive industry include wheel hubs, brackets, main covers, differential cases, steering knuckles, and other mechanical power transmission equipment.

Table 1. Typical properties of various pearlitic cast irons^[8,10]

Property	Nodular iron	Grey iron	Compacted iron
Tensile strength (MPa)	750	250	450
Youngs Modulus (GPa)	160	105	145
Elongation (%)	5	0	1.5
Thermal conductivity (W/mK)	28	48	37
Relative damping capacity	0.22	1	0.35
Hardness (BH 10/3,000)	217–255	179–202	217–241
Fatigue resistance (MPa)	250	110	200

A major challenge of using cast iron materials in many automotive applications is their propensity

to corrosion when they are in constant contact with various corrosive media under harsh service conditions. Hence, automotive components are usually coated to enhance their corrosion resistance. Several coating technologies have been applied to automobile parts to improve their service life^[5,11–13]. When the functionality of parts and threaded fasteners depends on certain fixed functions, such as the torque-tension relationship, phosphate coating is a prescribed process. The crucial procedure of phosphate-coating metal surfaces provides corrosion protection and lays the groundwork for strong paint adhesion. The most prevalent variation is the trication (Zn/Ni/Mn) phosphating procedure. The treatment involves dissolving the top surface layer via a combination spray-dip procedure. The substrate is dipped into a solution of phosphoric acid and metal ions, such as zinc, manganese, and nickel. An accelerator, such as nitrate, is introduced to the reaction that is reduced at the cathode. In addition, to nitrate reduction, other reactions include metal oxidation at the anode and hydrogen reduction at the cathode^[14]. Zinc phosphates are usually used as the coating material on truck wheel hubs at the final stage of production. This surface treatment process is purposely conducted to protect the component against corrosion. The phosphate coating crystals should cover the whole surface of the substrate and be uniformly distributed over the substrate's surface to ensure full protection. Different crystals are formed depending on the substrate and the most common ones are phosphophyllite and hopeite. The coating formation is initiated on nucleation sites on the substrate^[15], which implies that the initial substrate's surface properties are a controlling factor that would influence the phosphate crystal morphology.

However, the purpose of this study is to examine the initial surface characteristics of the machined and unmachined surfaces of nodular cast iron substrates and their impact on the zinc phosphate coating morphology; and consequently, relate the coating morphology to the general corrosion behaviour of the zinc phosphate and electro-coated (BASF cathoguard 570F black) ductile cast iron substrates. The study will also observe any impurity on the initial surface of the substrates that may affect the phosphate coating.

2. Materials and methods

Two truck wheel hubs were supplied by the Automotive Components Floby, Sweden, from where samples were extracted for this investigation. One of the wheel hubs was in as-machined condition while the other was already phosphate-coated as shown in **Figure 2**. Two samples for surface analysis were obtained from the machined and unmachined areas of the uncoated and zinc phosphate-coated hubs. These samples were provided in **Figure 3**, namely, 1) the unmachined sample, 2) the machined sample, 3) the unmachined phosphate coated, and 4) the machined phosphate coated. A metallographic sample was extracted from **Figure 2a** and prepared according to the standard metallographic procedure and etched in 4% Nital to reveal the pearlitic microstructure of the cast iron. The chemical composition (%-wt) of this material is as followed – 3.40% C, 2.39% Si, 0.27% Mn, 0.047% Mg, 0.030% P, 0.002% S, 0.30% Cu, and the balance is Fe. All the samples were characterized using an optical microscope as well as a scanning electron microscope coupled with energy-dispersive X-ray spectroscopy (SEM-EDS).

After the machining operation, the wheel hubs were pretreated with zinc phosphate coating followed by an electro-coating process using a BASF cathoguard 570F black reagent. The appearance of the component becomes black at the end of the coating process as observed in **Figure 2b**. Corrosion coupons were extracted from the coated hub (**Figure 2b**) and used for the assessment of the general corrosion of the hub according to the ASTM B117 salt spray standard procedure^[16]. The corrosion coupons were subjected to a six-week accelerated salt spray corrosion test. The corrosion-tested coupons were pictorially evaluated

according to the ISO 4628-3 standard^[17] when rating the degree of rust formed on parts that have been tested or used under accelerated or atmospheric conditions. The standard ISO 4628-3 rust rating scale is presented in **Table 2**.

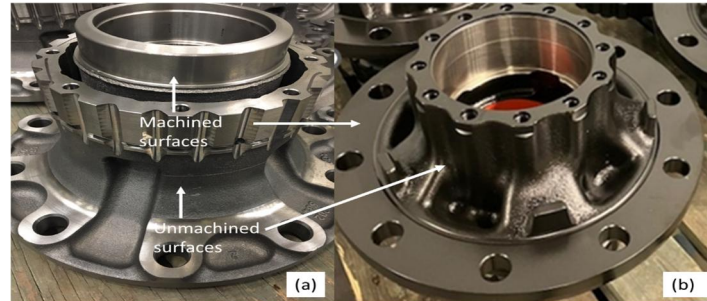


Figure 2. The as-machined (a) and phosphate-coated (b) truck wheel hubs.

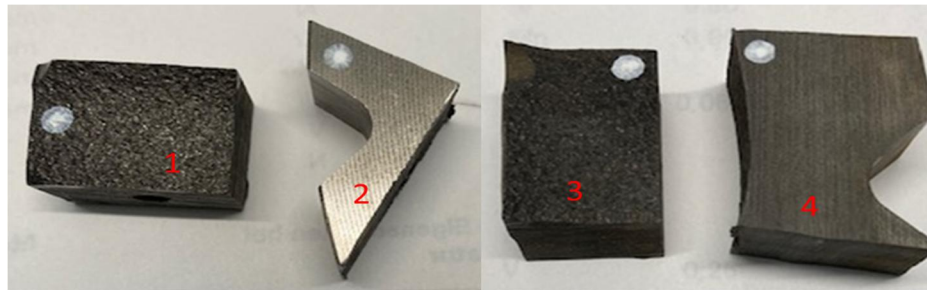


Figure 3. Cut out samples from the hubs: 1) the unmachined area, 2) the machined area, 3) the unmachined phosphate-coated region, and 4) the machined phosphate-coated area.

Table 2. The ISO 4628-3 rust rating scale

Degree	Rusted area (%)
Ri 0	0
Ri 1	0.005
Ri 2	0.5
Ri 3	1
Ri 4	8
Ri 5	40/59

3. Results and discussion

3.1. Microstructure analysis of the substrate

Figure 4 depicts the substrate's optical images and scanning electron micrographs. It can be seen from **Figures 4a** and **b** that the substrate consists of graphite nodules with a size less than and equal to 6. The graphite nodules are surrounded by ferrite and the matrix is mainly pearlite.

3.2. Surface analysis of substrates

The as-cast and machined surfaces of the cast iron substrates were analyzed to identify the composition of their surfaces. The purpose of this examination is to understand the impact of the substrate's surface characteristics influences the phosphate coating's effectiveness. **Figure 5** represents the images of the

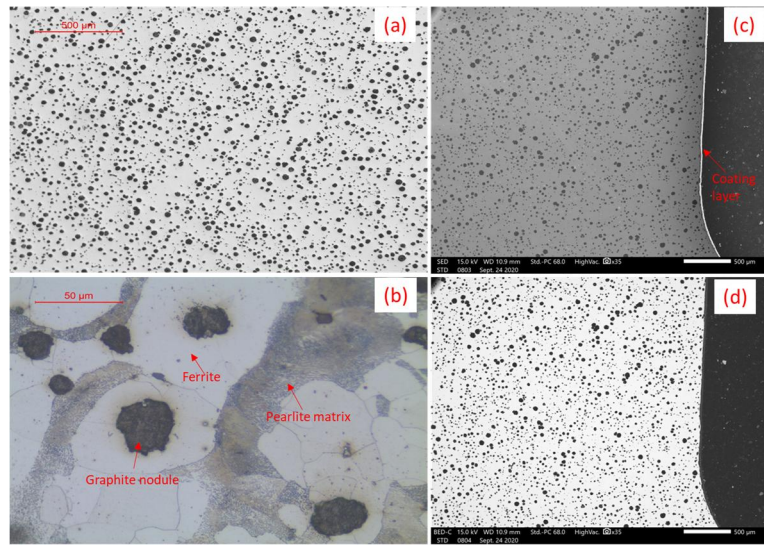


Figure 4. Optical micrographs of the substrate: (a) unetched, (b) etched, and SEM micrographs of electro-coated substrate cross-section (c) secondary electron and (d) backscattered image.

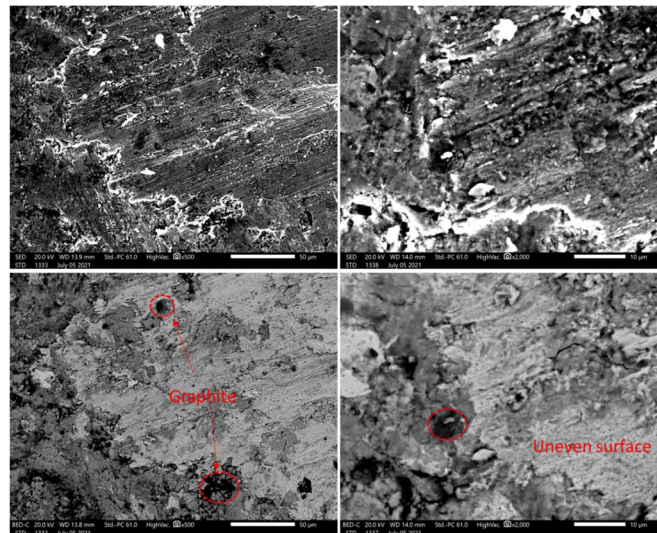


Figure 5. Micrographs of the unmachined surface of the cast iron substrate.

as-cast skin of the substrate while **Figure 6** provides the micrographs of the machined surface. It is apparent from **Figure 5** that the topography of the as-cast surface is uneven with different structural contrast. The surface of the as-cast substrate looks rougher than the machined one with some microcracks. The machined surface looks smoother, but cracks and rough surfaces were noted in some areas during the machining operation as seen in **Figure 6**. The graphite nodules are also observable on the machined and unmachined surfaces (the circled dark spots in **Figures 5** and **6**). Examination of **Figure 7** reveals that the as-cast surface contains different oxide compounds which include aluminium oxide, magnesium oxide, silicon oxide, iron oxide, and graphite. But in the machined surface, only Fe, Si, and C are detected (**Figure 8**). Some of these notable features and compounds may affect the deposition of phosphate coating on the substrate.

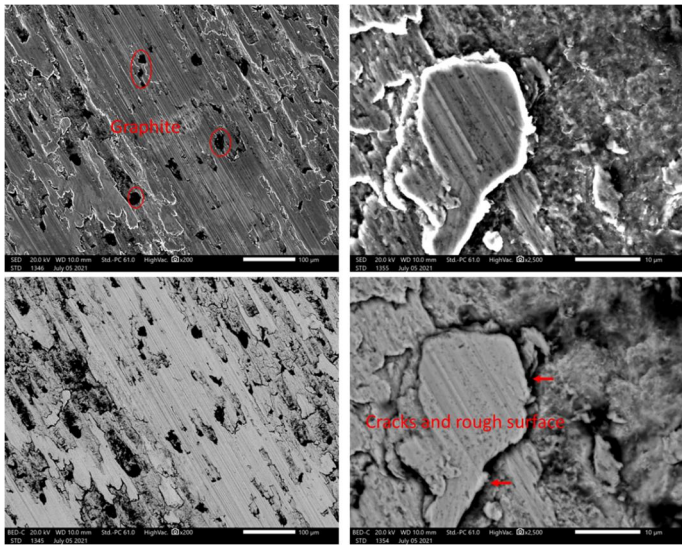


Figure 6. Micrographs of the machined surface of the cast iron substrate.

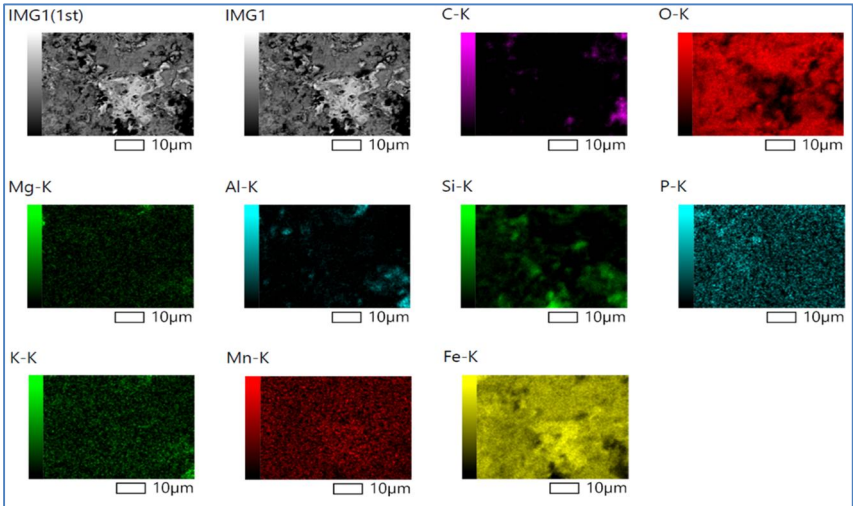


Figure 7. SEM-EDS elemental mapping of the unmachined substrate.

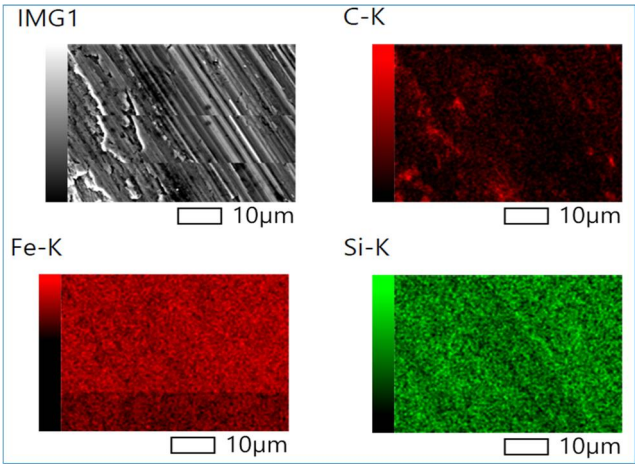


Figure 8. SEM-EDS elemental mapping of the machined substrate.

3.3. Surface analysis of the phosphate-coated substrates

Figures 9 and **10** show the SEM images of the phosphate-coated unmachined and machined nodular cast iron substrates, respectively. **Figure 9a** and **c** reveals some unmachined areas without coating, which the arrows indicate. Cracks are also noticed on the coated surface (**Figure 9a** and **b**). Similarly, from **Figure 10**, some uncoated regions are seen which are indicated by the arrows and broken circles (**Figure 10a** and **b**). Some rough coating areas are noticed in **Figure 10c**, which coincide with the machined surface with cracks and roughness, as shown in **Figure 6**. Besides, some regions with full coating coverage are observed in both the unmachined and machined surfaces of the coated substrates, see **Figures 9d** and **10d**.

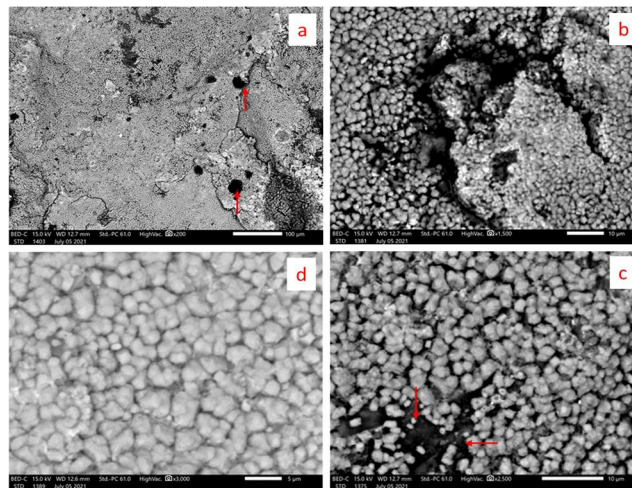


Figure 9. SEM micrographs of the phosphate-coated unmachined substrate.

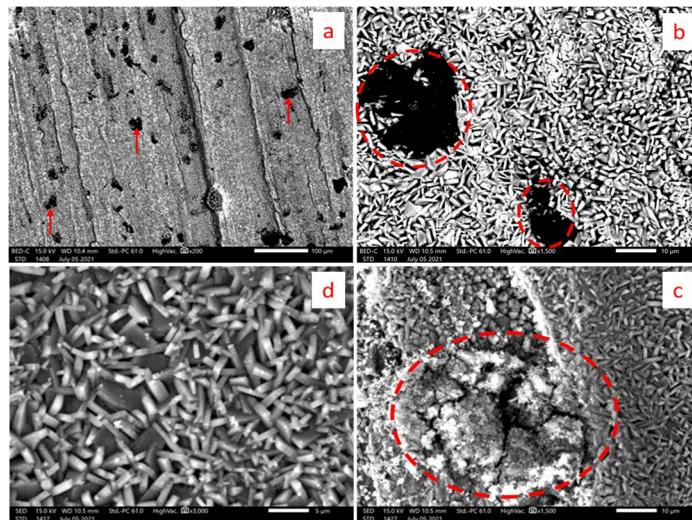


Figure 10. SEM micrographs of the phosphate-coated machined substrate.

Figures 11 and **12** represent the SEM-EDS elemental mapping of the phosphate-coated unmachined and machined substrates. It is indicative from **Figures 11** and **12** that, the regions with poor phosphate coating are rich in carbon, silicon, potassium, and aluminium. The presence of these elements suggests the existence of sand inclusions on the as-cast surface of the substrate (**Figure 11**). In **Figure 12b**, the regions with poor coating are the areas with graphite phases. It is also observed that the nature of the substrate surface affects the shape

and size of the phosphate coating. The unmachined surface with the as-cast skin shows spherical phosphate coating crystals (**Figure 9d**) while the machined surface reveals a long tetragonal crystal structure (**Figure 10d**). The average phosphate crystal size was measured and provided in **Table 3**. The crystal size of the unmachined phosphate-coated surfaces ranges from 0.633 μm to 3.182 μm , while that of the machined surfaces ranges from 0.828 to 6.240 μm . In summary, it is apparent from this investigation that, the presence of graphite and foreign inclusions adhesion on the substrate surfaces damages the phosphate coating. Also, rough surfaces with cracks lead to poor phosphate coating. The phosphate coating sizes and morphology are influenced by the surface characteristics of the coated substrate.

Table 3. Phosphate coating crystal size

Substrate	Mean crystal size (μm)	Min. crystal size (μm)	Max. crystal size (μm)	Standard deviation (μm)
Unmachined	1.74	0.63	3.18	0.50
Machined	2.58	0.83	6.24	0.98

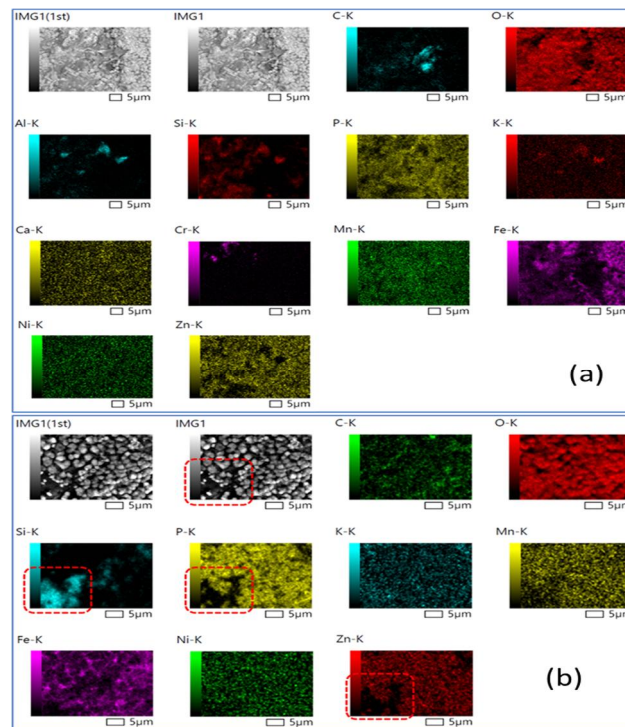


Figure 11. SEM-EDS elemental mapping of the phosphate-coated unmachined substrate.

3.4. Corrosion resistance of the phosphate-coated substrates

Based on the automotive application and the harsh service environment of the coated wheel hubs, the component is usually subjected to a six-week accelerated corrosion test by the ASTM B117 salt spray standard procedure^[16]. Before the salt spray corrosion test, scribe lines were created on the unmachined and machined surfaces of the test coupons. This is purposely done to simulate damages of the coating to assess the corrosion resistance of the coated wheel hubs after their exposure to a corrosive environment for a prolonged period. The electro-coat film thickness on the machined surfaces ranges between 22 μm and 30 μm .

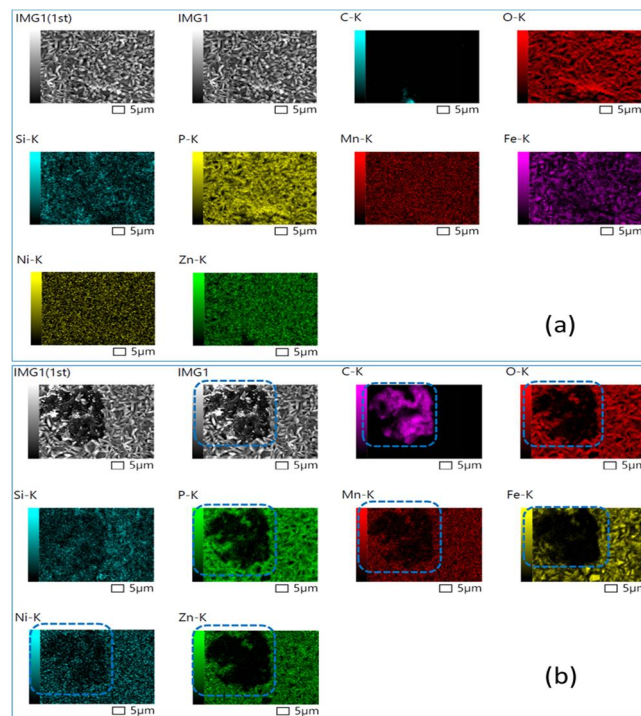


Figure 12. SEM-EDS elemental mapping of the phosphate-coated machined substrate.

The appearance of the coated wheel hubs before the corrosion test is displayed in **Figure 13**. However, at the end of their exposure to the corrosive media, the corroded coated test coupons were assessed using the standard procedures for evaluating the painted or coated specimens subjected to corrosive environments.



Figure 13. The appearance of the corrosion coupons before the test, where the solid and broken lines represent the unmachined and machined surfaces, respectively.

Figure 14 shows the pictures of some of the coupon specimens after the corrosion test. The inner corners of the unmachined surface of the wheel hubs were much more corroded than the outer surfaces of the unmachined region. The machined surfaces of the components are free of rust, implying excellent corrosion resistance. The corrosion performance of the coatings on the surface of the wheel hubs after their exposure to the corrosive medium was evaluated by the ASTM D1654 standardized procedure^[18]. This standard describes the scribing of coated test coupons into the substrate from the coating layer using one of several scribe tools. Analyzing the corrosion that occurred close to the scribed line allows for a qualitative evaluation of the coating's corrosion resistance. An estimation of the coating's corrosion resistance can be made using this

method, but it does not reveal anything about the mechanisms that prevent corrosion^[19]. The coated substrate is intentionally scribed (damaged) to expose the coating/metal interface to corrosive media to assess the function of passivating agents (if any), the level of coating adhesion to the substrate, and the surface state of oxides on which the coating is applied^[19]. Threadlike filaments may emerge when scribes are exposed to corrosive environments. The developed filaments can be assessed and scored according to the ASTM D1654 technique for corrosion testing of metallic and other inorganic coatings on metallic surfaces. In addition, the general corrosion resistance of the coated-wheel hubs exposed to the accelerated cyclic corrosion test procedures was evaluated and rated according to the ISO 4628-3 standard^[17]. Based on these standard methods, the corrosion resistance of the coated substrates was examined and scored as presented in **Table 4**. For this component to pass the corrosion resistance requirements, the width of the scribe lines after post-exposure to corrosive environments should be less than or equal to 8 mm. The general corrosion resistance of the entire surface of the coated substrates after the corrosion test is rated and must be scored a grade less than or equal to Ri 1 according to the ISO 4628-3 rust rating scale. As observed in **Table 4**, all five samples tested passed the scribed coated substrate corrosion resistance requirements. The coated machined surfaces of the wheel hub substrates also passed the general corrosion resistance requirements, but most of the unmachined surfaces of the coated substrate did not fulfil the requirement. In general terms, the corrosion resistance of the coated unmachined surfaces was disapproved according to the application requirements of the component. The poor corrosion resistance of the unmachined surfaces of the coated substrate could partly be influenced by the characteristics of the surface and the coating process parameters. However, to improve the surface properties of the unmachined areas, the cast component should be properly shot-blasted to remove any adhere foreign materials on the as-cast skin surface, which may hinder the adherence of the coatings on the substrate surface.



Figure 14. The appearance of the sample coupons after six weeks of accelerated corrosion test, where the solid and broken lines represent the unmachined and machined surfaces, respectively, and broken circles indicate the scribed lines.

Table 4. Post-corrosion evaluation of the coated parts based on the ASTM D1654 and ISO 4628-3 standards^[17,18].

Surface appearance	Scribe lines corrosion width requirement ≤ 8 mm		General corrosion requirement $\leq$ Ri 1 (see Table 2)		
	Machined	Unmachined	Machined	Unmachined	
				Outer area	Inner area
Sample 1	2.0 mm	2.0 mm	Ri 1	Ri 1-2	Ri 3
Sample 2	2.0 mm	2.5 mm	Ri 1	Ri 1-2	Ri 1
Sample 3	2.5 mm	2.0 mm	Ri 1	Ri 3	Ri 4
Sample 4	2.0 mm	2.0 mm	Ri 1	Ri 1-2	Ri 0
Sample 5	2.0 mm	2.0 mm	Ri 1	Ri 4	Ri 4

4. Conclusions

This investigation examined the surface characteristics of machined and unmachined surfaces of ductile cast iron wheel hubs. The influence of these surface imperfections on the Zinc phosphate coating and the subsequent corrosion resistance was assessed and rated according to the standard procedures. Surface analysis of the machined and unmachined hubs revealed the existence of graphite and sand bonding on the substrate surface, possibly affecting the phosphate coating. Foreign material on the as-cast surfaces and cracks caused poor phosphate coating. The average phosphate coating crystal size is 1.742 μm and 2.578 μm for the as-cast and machined substrates, respectively. The substrate surface roughness influences the size and shape of the phosphate coating crystals. It is recommended that the as-cast surface should be properly cleaned to remove sand bonding on the as-cast skin. Surface cracks on the machined surface should be minimised to ensure perfect phosphate coating. Generally, the corrosion resistance of the coated unmachined surfaces was disapproved based on the application requirements of the wheel hubs. The poor corrosion resistance of the unmachined surfaces of the coated substrate was attributed to the influence of the substrates' surface characteristics on the coating processes. The cast surfaces should be properly shot-blasted to remove any adhere foreign materials on the cast skin to enhance the adhesion of the phosphate coatings on the substrate surface.

Acknowledgments

The author appreciates the efforts of his colleagues at the Production Unit, Automotive Components Floby AB, for providing the process information and the components used for this investigation.

Conflicts of interest

The author declares no conflicts of interest.

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