

Surface analysis of conversion coating of ASTM A 516

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Abstract

Surface engineering is a vital aspect of manufacturing industries owing to its benefits both in surface protection and aesthetics. It has been extensively used in various industries to guard against corrosion which is a naturally occurring and highly undesirable phenomenon. Present research has endeavored to analyze protection of ASTM A516 (Grade 70) from corrosion through surface engineering. Different methods of surface treatment and conversion coating were carried out to efficiently enhance corrosive protection. Comparative analysis of various samples was conducted to analyze their ability to resist corrosion. Samples with surface treatment followed by conversion coating were found to be effective even against 0.7% aqueous sulfuric acid with no significant cracks in the coating layer. On the other hand, conversion coated only samples showed protection against 0.35% acid. The coating of conversion coated only samples was found to have gaps/ cracks as indicated by 3% Cupric Sulfate whereas no such gaps were found in surface treated samples. Optical microscopy identified a more uniform coating thickness for surface treated samples in comparison with conversion coated only samples. In depth morphology analysis using SEM highlighted that surface treated samples had low porosity preventing the corrosion elements to reach the substrate thereby implementing higher corrosion potential.

Keywords: ASTM A 516 (Grade 70); Corrosion; Surface engineering; Conversion coating

1. Introduction

Corrosion is a highly undesirable phenomenon which occurs naturally at varying rates depending upon the availability of mainly basic and then catalyzing agents [1][2]

Nomenclature

EDS	Energy Dispersive X-ray Spectroscopy
RPM	Revolutions per Min
SEM	Scanning Electron Microscope

Corrosion of iron and steel is known as rusting which produces Fe_2O_3 (rust) as final product. The overall process comprises of a number of reaction, collectively as redox reactions [3]. Corrosion of carbon steel causes physical degradation which, annually, results in huge financial losses worldwide [4]. Coatings are an effective method of enhancing corrosion resistance for various materials [5]. It can serve the dual purpose of surface protection and aesthetics [6]. Various type of coating technologies are used worldwide [7]–[9]. Conversion coating is a extensively used process owing to its simple and economical procedure as well as enhanced protection [10]. There are different types of conversion coating processes with varying effectiveness mainly depending upon the treatment solution and process time, among other factors. Past researchers have used different methods of

conversion coatings to analyze their resistance potential. In few instances, surface was mechanically treated in combination with coating to enhance the barrier towards corrosion. Zeng et al [11] concluded that phosphate coating not only improves the biocompatibility of We43 alloy but also adds to the corrosion resistance. In another related study, it was found that effectiveness of phosphating can be further improved by adding benzotriazole [12]. Jinlong et al [5] investigated the corrosion resistance of NiTi shape memory alloy in NaCl, H_2SO_4 and borate buffer solution. The corrosion protection of different samples was attributed to donor concentration in conversion coating and film thickness. Ghaziof et al [13] studied the effects of chromium carbon coating in combination with mechanical and electro polishing procedures. It was found that steel gets a high corrosion barrier as a result of coating. The pre-coating surface treatment ensured a defect free coating thereby improving the protection against corrosion. Ganesh et al [14] carried out thermo-mechanical treatment on SAE 304 stainless steel. XRD analysis highlighted the enhanced inter granular corrosion resistance and materials potential against sensitization. In another related research, Onofre et al [15] found that in addition to corrosion protection, conversion coating enhanced the adhesiveness effect. Literature also highlights that Magnetite coating (Fe_3O_4) have been researched on

different materials using various methods[16]. Nagode et al. [17] deposited magnetite coating on grey cast iron plates which was found to be stable at elevated temperatures. Possibility of formation of hematite was also observed owing to the oxidation of magnetite layer. A similar coating was made on cast iron in the presence of chromium and silicon by Arab and Rahimi [18]. It was concluded that the resulting magnetite coatings was able to withstand 150 hours of salt spray test. In another related study [19], carbon steel was coated with magnetite under varying temperature, voltage and electrolytic composition conditions. Resultantly, coating thickness was found to be a function of input parameters.

2. Design of experiment

2.1 Specimen material

Optical emission spectroscopy of the selected specimen (ASTM A 516 grade 70) material was carried out with results given in Table 1. ASTM A 516 was selected owing to its worldwide usage in various industries. Its superior mechanical properties [20] as shown in Table 2, makes it a preferred choice specially in construction sector.

Table 1. ASTM A516 - Chemical Composition

Element	Percentage
Mn	0.85 – 1.2%
Si	0.15 – 0.40%
C	0.10 – 0.28%
P	0.010 – 0.035%
S	0.010 – 0.035%
Fe	balance

Table 2 ASTM A516 - Mechanical properties [20]

Property	Value
Tensile Strength	511 N/mm ²
Yield Strength	424 N/mm ²
Elongation	24%

2.2 Sample preparation

Work specimen was cut into samples size of 10 inches' length and 6 inches' width, having a thickness of 0.12 inches'. Two different batches were made for comparison. The samples in each batch were given separate treatment as per the categorization shown in Table 3. Type 1 samples were given hot alkaline bath. During this process, these samples were kept for 45 minutes in aqueous mixture of sodium nitrate (NaNO₃) and sodium hydroxide (NaOH) in a ratio of 1:3 at 130°C. As a result, samples were coated with coating of magnetite (Fe₃O₄). Type 2 samples were first surface treated by grinding them with emery paper grinder (grit size - 300 μm) at 3000 RPM. These samples then underwent conversion coating process with hot

alkaline bath, as explained earlier. These samples are as shown in Fig. 1.

Table 3 Specimen types

Specimen	Surface Treatment	Conversion Coating
Type 1	✗	✓
Type 2	✓	✓

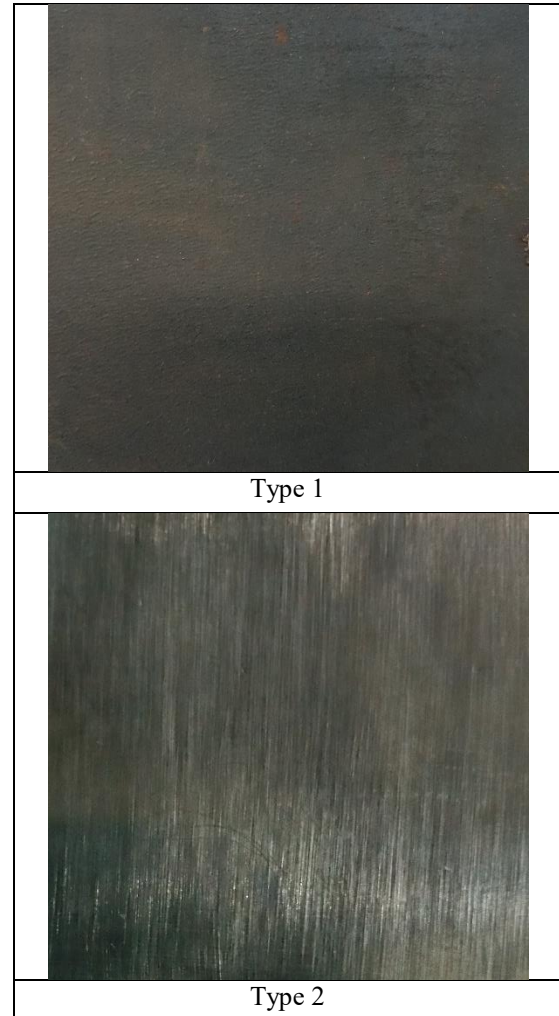


Fig. 1 Types of samples

3. Coating tests

Magnetite coating was tested for its corrosion resistance and uniformity using stability and continuity test respectively. Literature indicates the use of sulfuric acid for stability test which acts as corrosion accelerator [21]. Various materials including Stainless Steel (type 304) [22], Titanium [23] and concrete [24] have been tested using such tests. Similarly, cupric sulfate solution has been used in the past to check uniformity of conversion coatings [25].

3.1 Stability test

Stability test was conducted to ascertain the effectiveness of the coating process. It signifies the

strength of coating under corrosive environment. Drops of 0.175% aqueous solution of sulfuric acid were put on the coated surface. The acid takes away any poor or low quality coatings on contact. It was found that neither of the coatings was washed away by the acid. In the next step stronger 0.35% solution was used which too was unable to dislodge the coatings. In the third stage 0.7% acidic solution removed coating from type 1 sample but type 2 coating kept intact. Results displayed in Fig. 2.

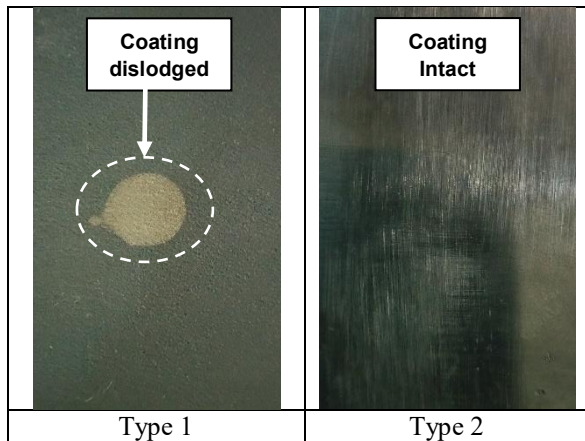


Fig. 2 Stability test

3.2 Continuity test

Continuity test was conducted to find the presence of gaps and cracks present in the coating. Such defects can result during the initial coating procedure. In this test 3% Cupric Sulfate solution drops were put on the sample surface for 30 seconds. Cracks or gaps in the coating were highlighted by red color copper deposits. Results are displayed in Fig. 3.

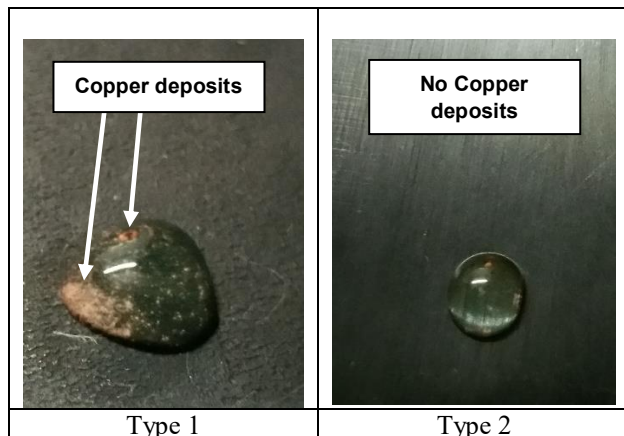


Fig. 3 Continuity test

Drops of cupric sulfate on type 1 sample reflected red color confirming the presence of copper deposits. It was indication of the presence of cracks and/or pores. However, no significant deposits were seen on type 2 samples. It was concluded that type 2 samples had continuous and defect free coating.

4. Surface analysis

4.1 Cross sectional analysis

Optical microscopy of the samples was conducted for further analysis of coating. The cross section of samples was observed to check the coatings for uniformity. Fig. 4 shows the cross sectional view of type 1 and type 2 samples.

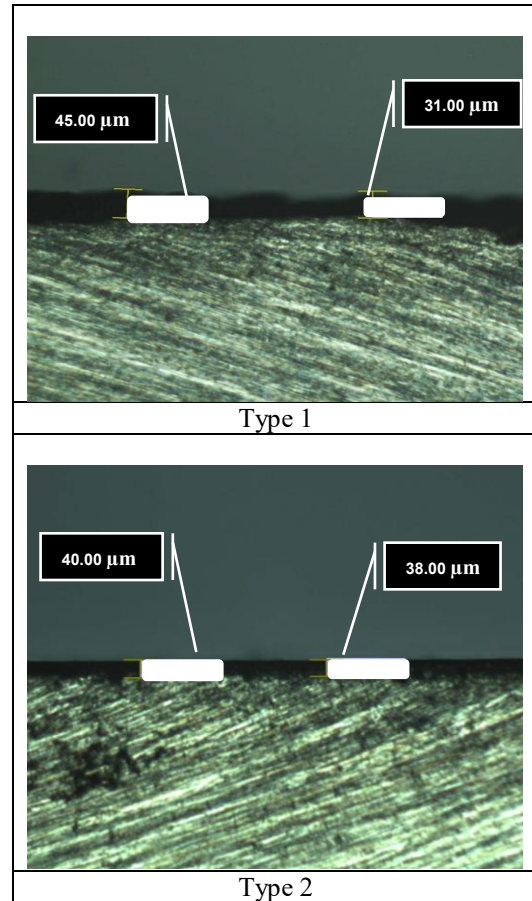


Fig. 4 Cross sectional view of samples

The images show that type 1 has a coating with varying thickness from 26 μm to 45 μm. The base surface is uneven with few sharp edges and corners. Coating thickness of type 2 sample was found to be more uniform with thickness from 38 μm to 44 μm. Substrate had a much leveled surface owing to the surface treatment carried out before conversion coating.

4.2 Morphology analysis

SEM analysis was carried out to observe the surface morphology of samples as shown in Fig. 5. Porosity on the surface allows corrosion ingredients to act effectively at surface and sub-surface levels to initiate and catalyze the rusting process. Type 1 showed high amount of porosity and unevenness. Porosity of type 2 sample was found to be much lower due to the grinding surface treatment.



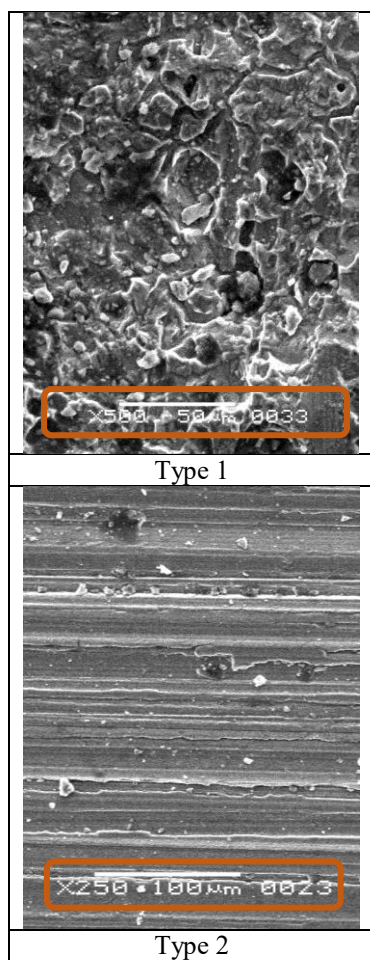


Fig. 5 SEM images

EDS analysis of type 1 sample indicated the presence of Fe_2O_3 (oxide known as rust) and other corrosion by products in addition to coating material Fe_3O_4 . In case of type 2 sample no corrosion elements were present whereas a uniform magnetite (Fe_3O_4) layer was detected.

5. Conclusion

Present research has chalked out the following conclusions:

- Conversion coating of magnetite (Fe_3O_4) is an effective way to protect against corrosion. The protection can be further enhanced by surface treatment through grinding prior to the coating process.
- Coating layer of magnetite (Fe_3O_4) displayed effective protection against up to 0.35% aqueous sulfuric acid solution in stability test. Coating on ground surface was found effective even against 0.7% acidic solution. SEM analysis revealed that surface grinding removes surface pores which otherwise are prone to initiate corrosion process.
- The coating of unground surface displayed some gaps in continuity tests using 3% Cupric Sulfate solution. However, no such gaps were present in the coating layer of ground samples. This is due to the uniform coating thickness of

surface treated samples varying from 38 μm to 44 μm as observed in Optical microscopy images. On the other hand, in case of untreated samples the coating thickness varied from 26 μm to 45 μm .

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