The Effects of Heat Treatment on Area Percent Porosity and Corrosion Behavior of High-Nickel Thermal Spray Coatings

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Abstract

Samples of two Thermal Spray Coating (TSC) alloys on a low carbon steel substrate were obtained: alloy C276 and Nicko-Shield 200. Specimens of each alloy were subjected to heat treatments at temperatures at 1100° for 60 minutes and 1200° for 10 minutes, with some specimens left in the as-sprayed condition. Three replicates were prepared for each condition. Thin 1" strips were cut using a diamond wafering blade for porosity measurements and 2" x 1" specimens were cut for corrosion testing using a SiC abrasive saw. The porosity specimens were mounted in acrylic resin, polished, and examined using SEM. From these images, area percent porosity was calculated using automatic image analysis software. The average porosity of the untreated specimens was 2.51% for C276 and 2.2% for Nicko-Shield 200; these values were not statistically different. Heat treatment resulted in no significant change in area percent porosity. The corrosion specimens were mounted in acrylic so that only the coating surface was exposed to the environment. These specimens were immersed in 20% H_2S0_4 at 65°C for 200 hrs. Each specimen was weighed before and after immersion to measure mass loss due to corrosion. The average mass loss of as-sprayed Nicko-Shield 200 was 6.405 mm/yr. The heat treated specimens of Nicko-Shield 200 showed improved corrosion resistance, with an average mass loss rate of 0.92 and 0.54 mm/year after treatments at 1100° for 60 minutes and 1200° for 10 minutes, respectively. C 276 failed corrosion testing under all conditions. Overall, heat treating was found to increase corrosion resistance without effecting porosity. Visual evidence suggests that the change in corrosion behavior was caused by the formation of an oxide layer during heat treatment.

Keywords: materials engineering, thermal spray coating, corrosion, porosity, sintering, heat treatment, Nickel alloy,

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Introduction

Statement of Purpose

Corrosion in Chevron's oil treatment pipelines is causing significant losses of time and money. The purpose of this project was to evaluate the performance of Ni-based thermal spray coatings (TSCs) developed by Scoperta on carbon steel substrates under conditions present in Chevron's oil treatment facilities. The corrosion behavior of the coatings was examined and compared to the performance of a conventional Ni-based thermal spray coating typically used under these operating conditions. In addition, the effects of various heat treatments on the porosity and corrosion behavior of the coatings were examined. This project was meant to assess the ability of Scoperta's coating to meet Chevron's demands and explore the possibility of improving TSC corrosion resistance through heat treatment.

Justification

A 1998 study conducted by members of NACE, U.S. Congress, and the Department of Transportation estimated the direct cost of corrosion damage in several sectors if the economy. The result: $137.9 billion, an astronomical figure representing 1.6% of the GDP[1]. In the oil industry alone, corrosion damage causes $7 billion worth of damage annually[2]. Clearly, corrosion is a problem that must be addressed. As a key player in the oil industry, Chevron is no stranger to corrosion problems. Oil and chemicals used in the refining process cause high corrosion rates in their carbon steel pipelines. This project will attempt to address this problem by evaluating the porosity and corrosion-resistance characteristics of Ni-based TSCs for use in highly corrosive environments.

Background

TSC History

The origins of thermal spray technology can be traced back to the early 1900s in Zurich. Dr. Max Ulrich Schoop is credited with the original design, which used high pressure gasses to direct molten metal through a series of hoses and through a spray nozzle. This process, though crude, was revolutionary for the time period, and proved to be very effective and superior to the then-current alternatives[3]. TSC technology has vastly improved since those early years—however, the basics remain the same. Depending on the particular spray process used, the coating material (usually a metal) may be in wire, powder, or rod form. The materials is heated and accelerated by high pressure gases toward the substrate. Upon impact, the spray particles "flatten and form thin platelets that conform and adhere to the irregularities of the prepared surface and to each other. They cool and accumulate, particle by particle, into a lamellar, cast-like structure"[4]. The process is shown in Figure 1.

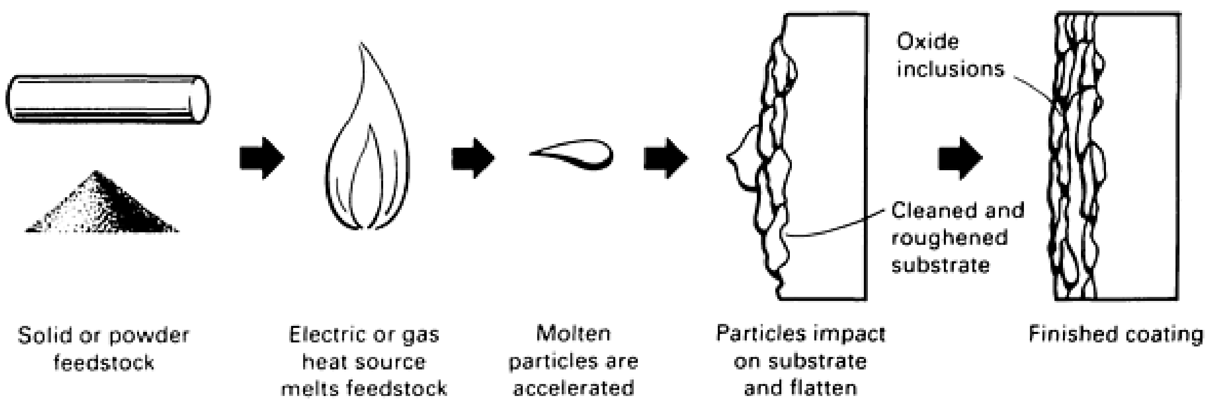

Figure 1: General TSC deposition process[4].

Properties

The main advantage of TSCs is their ability to confer enhanced properties to the surfaces of inexpensive substrates. A variety of coating materials can be applied via thermal spray methods depending on the desired properties of the coating. It should be noted, however, that as result of the spraying process, TSCs generally have properties that differ from those of the bulk

material. Oxides, porosity, unmelted particles, and other impurities are often present to some degree in the finished coating, as seen in Figure 2.

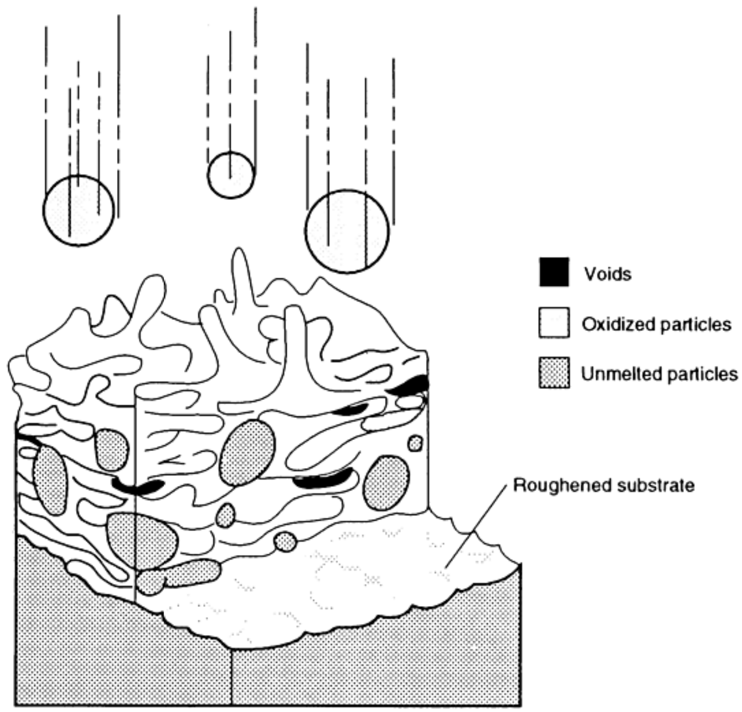

Figure 2: All thermal spray processes lead to a degree of voids, oxides and unmelted particles[4].

Controlling these impurities and defects—and, in turn, the properties of the coating-- is accomplished by varying process parameters. The type of spray process used can significantly affect the properties of adhesion strength, thermal shock resistance, abrasion resistance, and corrosion resistance. Although several of these properties conflict with each other, the coating can be optimized for its application[5].

The property with which falls within the scope of this project is corrosion resistance, which is closely linked to the porosity of the coating. Porosity can be influenced by particle velocity and particle temperature. When a coating particle, or "splat," impacts the substrate, the degree to which it flattens and spreads depends on its impact velocity. At higher velocities, the splat will flatten more fully, leading to more even coverage and less porosity. Temperature is

also a key parameter. The particles must become sufficiently hot to melt and adhere to the substrate upon impact. However, operating at high temperatures can lead to oxide formation and possible metallurgical changes to the substrate. A balance must be struck to ensure optimal properties. Depending on the specific process used, TSC porosity ranges from 2% to 17%[4].

The coatings that are the subject of this project were deposited using the Twin Wire Arc spraying technique. In this process, two consumable wires are fed automatically to meet at a point in an atomizing gas stream. An electrical arc is truck across the wire electrodes and melts the tips of both wires. The atomizing gas is directed across the arc zone, shearing the molten part of the wires to form the atomized spray[6]. A Schematic of this process is shown in Figure 3. TWA coatings typically have porosity of between 3% and 8%[7].

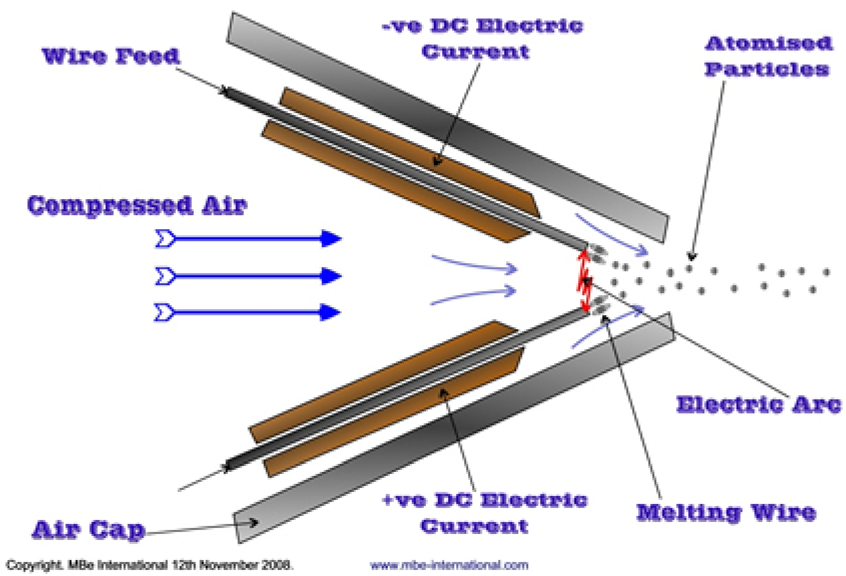

Figure 3: The Twin Wire Arc Spray Process is used by Scoperta to deposit their TSC alloy[8].

Scoperta and Chevron

Scoperta Inc. has developed several Ni-based coatings for use in Chevron's facilities. The coatings were developed to confer corrosive resistance properties to pipeline which will be in

contact with several corrosive substances, including hot sulfuric acid. The pertinent property for Chevron's purposes is porosity, as it is directly related to the corrosion resistance of the coating. Though porosity can be influenced by the application technique, studies have shown that heat treatment can also have an effect. Included within the scope of this project is the determination of specific heat treatment parameters that will lower the coating's porosity and improve its corrosion resistance.

Porosity and Heat Treatment

Due to the nature of the thermal spray process, all thermal spray coatings inherently contain some level of porosity. This porosity is detrimental to the effectiveness of thermal spray coatings used for corrosion resistance applications due to the increased surface area exposed to the corrosive environment. In addition, interconnected pores connected to the surface can allow corrosive material to penetrate the surface of the coating and thereby reducing its effectiveness.

The hypothesis of this experiment is that, through a mechanism similar to that seen in sintering operations (Figure 4), the porosity of the thermal spray coating may be reduced through high temperature heat treatments. In a sintering process metal or ceramic particles are raised to an elevated temperature where through diffusion driven by the reduction of surface energy, the spaces between particles (pores) reduce in size leading to an overall decrease in porosity[5]. One goal of this project is to attempt to increase the corrosion resistance of a thermal spray coating through a sintering-like reduction of porosity and to compare various heat treatments to achieve maximum porosity reduction and corrosion resistance.

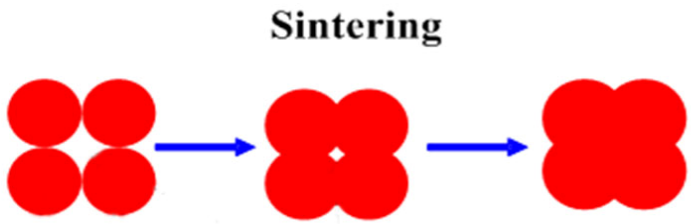

Figure 4: Schematic showing the reduction of porosity in a sintering operation.

Corrosion

Sulfuric acid (H_2SO_4), a harshly corrosive substance, is used as a catalyst in a chemical reaction to produce large volume, large value products at Chevron's refining facilities. This acid is typically used at a high concentration (> 90%); however, this is not a great concern because low carbon steel is resistant to corrosion in concentrated sulfuric acid. Problems arise, however, when steel come in contact with hot, dilute H_2SO_4. To combat this corrosion problem, the use of TSCs is being investigated as a low-cost solution.

TSCs are desirable in these situations due to their ability to confer enhanced corrosion protection to inexpensive substrates, such as low carbon steel. Nickel and its alloys are commonly used for these applications due to their superb corrosion resistance. The TSCs that this project focuses on are Nickel-based, with high amounts of Chromium and Molybdenum, as well as small amounts of other elements. The addition of alloying elements supplements the already impressive corrosion resistance of Nickel. The additions of Chromium and Molybdenum make the resulting alloy especially resistant to H_2SO_4 at low concentrations, making these alloys ideally suited for Chevron's needs[9].

Broader Impacts

Chevron

The primary stakeholder in this project is the Chevron Corporation. As the end-user, Chevron has a specific problem: high corrosion rates in its carbon steel pipelines, caused by oil and its byproducts. There are several ways to address this problem: use an inexpensive material

and plan for frequent maintenance, use an expensive, corrosion resistant material, or overlay a cheap material with a more resistant alloy. Thermal spray coatings offer all the advantages of the latter option without the drawbacks of welding. TSC processes are ideally suited for the petroleum industry due to the "speed, portability, and low heat input involved…Also, the advantages of a thinner coating without base metal dilution or heat-affected zones are obvious."[10]

Evaluation of the corrosion-resistance and adhesion characteristics of Ni-based TSCs is required to aid the development of a coating that can withstand continuous exposure to corrosive substances. Successful evaluation of these coatings is critical to the company; the results of this project could lead to the development of various new coatings and ultimately solve Chevron's corrosion problem. Data and analysis from our project will give a better understanding of the corrosion characteristics of the coating and substrate and thus will satisfy Chevron's needs. In addition, the experimental procedure performed for this project can serve as an example for future testing, since no ASTM standards exist for the specific tests conducted.

Stakeholders

Clearly, there are alternative solutions to Chevron's corrosion problem that do not involve the development of corrosion-resistant coatings. However, the decision to pursue this option depends heavily upon economic factors, on which the profitability and future success of Chevron hinges. The success of this project could result in considerable savings in material cost over many years. It is estimated that corrosion damage accounts for 60% of all maintenance costs in the oil industry[1]. The decrease in corrosion would likely reduce the frequency of corrosion related spills and failures, which have associated clean-up costs. The resulting decrease in expenses would lead to increased revenue, satisfying the needs of the company's shareholders. A better understanding of the corrosion characteristics of Ni-based thermal sprays will satisfy these needs.

Society

In a broader perspective, the success of this project could have ramifications on the entire oil industry. The knowledge gained through this project could eventually lead to a shift in the oil industry toward Ni-based thermal sprays. Of course, intellectual property and other legal issues may slow or even halt this shift, but the big picture is that the industry has a need for a corrosion-resistant coating, and the success of this project may eventually fulfill that need. The growing interest in TSCs may also lead to the improvement of coating technology and the understanding of their properties. This can include coating techniques, alloys, and heat treatments, and how these aspects affect the coatings mechanical and corrosive behavior.

Experimental Procedure

Factors

Testing was conducted on two different TSC alloys to determine the effect of composition on corrosion behavior. The first alloy is designated Hastelloy C276, an extremely versatile and corrosion resistant alloy. C276 is commonly applied via TSC techniques for various applications that require good corrosion resistance. The second alloy is designated Nicko-Shield 200, an alloy of proprietary composition developed by Scoperta. Both alloys are Nickel-based with high amounts of Molybdenum and Chromium. However, since the exact compositions are not known (due to proprietary reasons) our analysis of the effect of composition was limited.

To investigate the effects of heat treatment on TSC porosity and corrosion behavior, several heat treatments were performed on specimens of each alloy. Heat treatments at temperatures of 1100°C and 1200°C, for 10 and 60 minutes were performed. These heat treatment parameters correspond to typical annealing temperatures for Nickel alloys, so they should have a similar effect on Nickel-based TSCs. In addition, untreated specimens of each

alloy were prepared—these will serve as a control group. All heat treatments were carried out in an inert environment courtesy of Bodycote Thermal Processing (Fremont, CA)

In total, five treatments were applied to each of two alloys, resulting in 10 separate groups. Three replicates of each group were performed, meaning 30 specimens were prepared for each test—porosity and corrosion. From the original 6” x 6” plates we received, each specimen was cut to the size dictated by the appropriate ASTM standard.

Porosity Measurements

Porosity measurements were carried out according to ASTM E2109, which calls for optical microscopy or SEM images and analysis using image analysis software. Thin cross-sectional slices one inch in length were cut using the LECO 34314 Diamond Blade Precision SAW. Each specimen is mounted in acrylic resin, after which metallographic procedure was performed according to ASTM 1920. Polishing is performed down to a particle size of 0.06 µm.

Image capture was performed using an SEM. Three images were taken of one section of coating. These images were spliced together to create one larger image that captures the entire thickness of the coating. This process is illustrated in Figure 1. This process was repeated five more times; as a result, a majority of the coating is imaged.

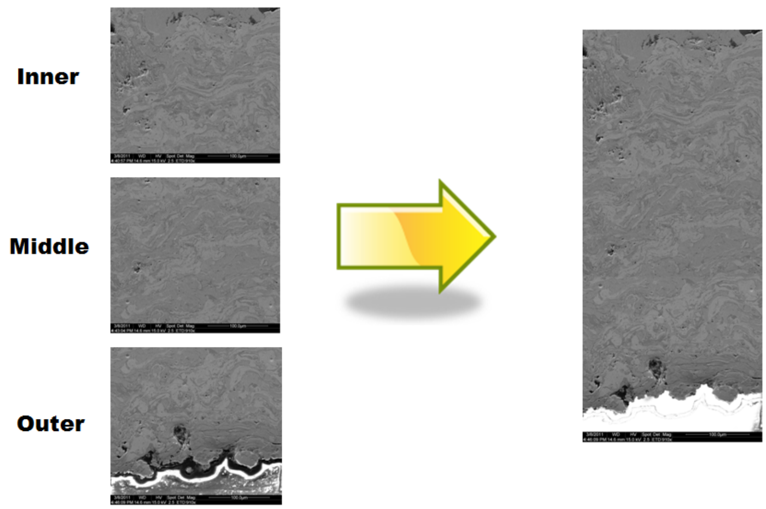

Figure 5: Several images were spliced together to create one image of the entire coating thickness.

Once the image collection was complete, the jpeg file was imported into ImagePro Express image analysis software. Color segmentation was performed to isolate porosity in the coating. Next, a histogram of color analysis was created, giving us the total area percentage represented by pores in the coating. These steps were performed for each large composite image, for a total of six values. This process is summarized in Figure 2.

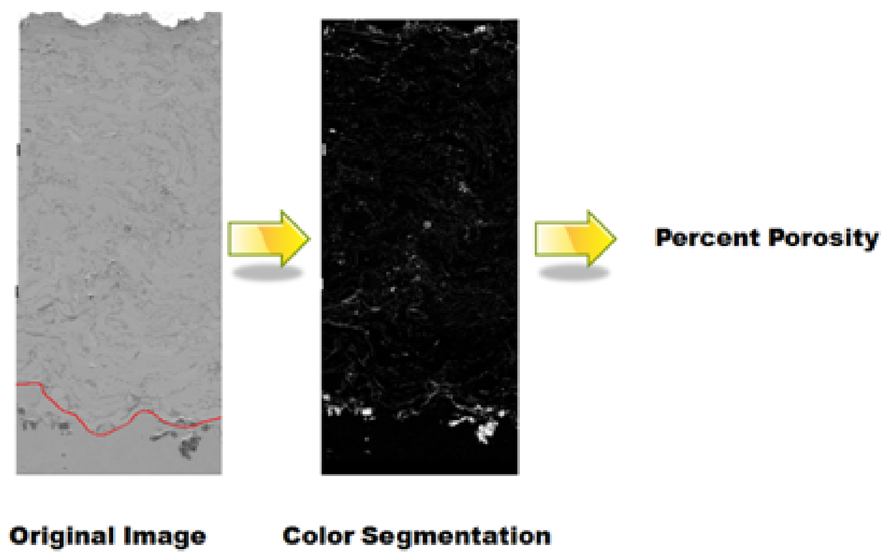

Figure 6: ImagePro software is used to segment the image and calculate percent porosity.

Corrosion Testing

In order to analyze the corrosion resistance of the two thermal spray coatings, three samples of each alloy at each heat treatment condition were subjected to a 200 hour immersion corrosion test in 20% sulfuric acid at 65°C. Corrosion testing was performed according to ASTM G 31 – 72. Due to the fact that the corrosion testing coupons were coated on only one side, the remaining sides of the samples were masked with acrylic in order to prevent exposure of the bare steel substrate to the sulfuric acid (Figure 7).

Figure 7: Corrosion test coupon masked with acrylic to prevent exposure of bare steel substrate to acid

Mass and exposed surface area measurements of each sample were recorded before immediately before the beginning of the corrosion testing. Each sample was placed in a separated sealed container and heated on a hot plate to 65± 5 °C. After 200 hours the samples were removed from the acid and cleaned of any corrosion produce then weighed to determine the total mass loss for corrosion rate calculations.

Results

Due to unforeseen delays, only heat treatments of 1200°C for 10 minutes and the 1100°C for 60 minutes were completed and available before the deadline for this project. Because of this no data was collected for the other two heat treatments.

Porosity

The area percent porosity of each of the heat treated and as-sprayed samples were measured using image analysis of cross sectional SEM micrographs. Each porosity measurement was taken across the entire thickness of the coating due to the non-uniform distribution of pores. Initial measurements of the two alloys, C276 and Nicko Shield 200, revealed that both alloys had average area percent porosities between 2% and 3% and could not be shown statistically to be different (Figure 8).

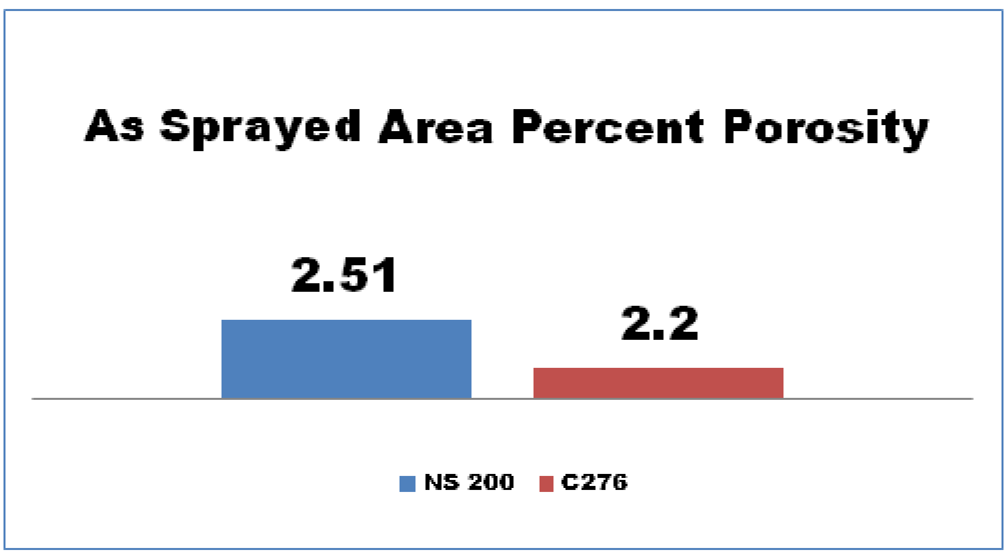

Figure 8: Average area percent values for as sprayed NS 200 and C276. The difference in porosity was found to be statistically insignificant.

As previously mentioned samples for heat treatments of 1200°C or 60 min and 1100°C for 10 min were not available before the deadline for this project. Because of this no correlation was able to be made between the individual factors of time and temperature with the porosity. Porosity data collected is shown in Figure 9 below. Each heat treatment condition was found to

have an average area percent porosity of approximately 2.5%. The percent porosities of the heat treated samples were not found to have any statistically significant difference from each other. In addition the heat treated samples were not significantly different from the as sprayed condition. The porosity data collected in this experiment suggests that heat treatments of 1200°C for 10 min and 1100°C for 60 min had no effect on the total area percent porosity.

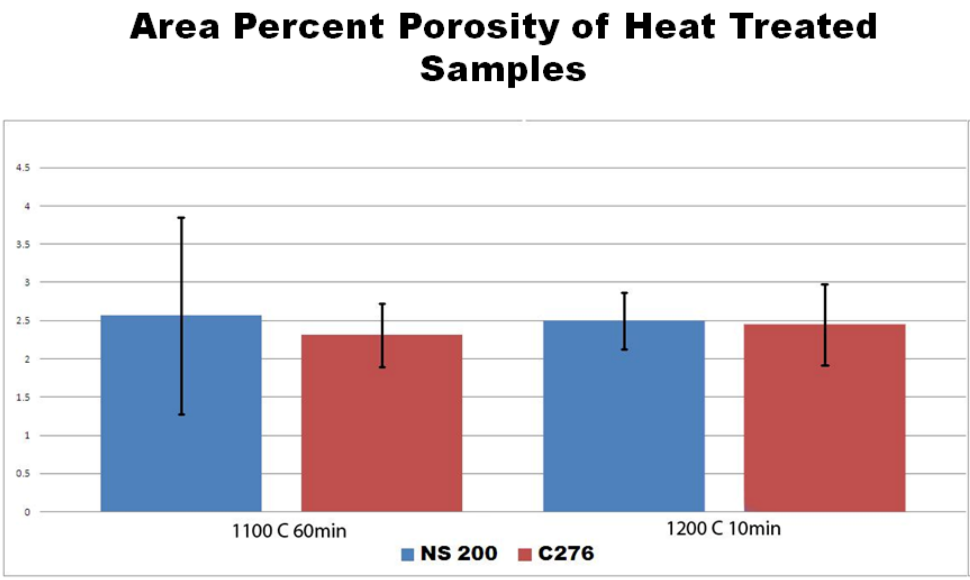

Figure 9: Plot of average area percent porosity of heat treated samples. No significant difference was found between any combination of alloy and heat treatment.

Corrosion

After completion of the corrosion tests, the specimens were removed from the acid and cleaned to remove any corrosion product that may have formed. Each sample was weighed; this mass measurement was compared to the mass before the test to determine the mass loss. The corrosion rate (mm/year) was calculated using Equation 1:

$$Corrosion\ Rate = \frac{K \times W}{A \times T \times D}$$

Where K is a rate constant, W is the mass loss of the specimen in g, A is the exposed surface area of the coating in cm^2, T is the time of exposure in hours, and D is the density of the coating in g/cm^3. Since the desired form of the corrosion rate is mm/year, $K = 8.74 \times 10^4$.

Upon completion of the corrosion tests, all specimens of C276 had been completely corroded away. The acid had destroyed the coating and had begun to attack the steel substrate (Figure 10). Any mass loss measurements would thus be measuring the mass of both the coating and the substrate, preventing an accurate corrosion rate from being calculated. A summary of the mass loss measurements and corrosion rate calculations for Nicko-Shield 200 is shown in Table I, and the averaged corrosion rates are shown in Figure 11.

Figure 10: All C276 specimens were corroded completely.

Table I: Corrosion rate calculation for Nicko-Shield 200.

Specimen	**Mass loss (g)**	**Surface Area (cm^2)**	**Corrosion Rate (mm/year)**
As Sprayed-1	4.28	13.05	16.56
As Sprayed-2	1.18	9.81	6.06
As Sprayed-3	1.38	9.81	7.08
1100°C 60 min-1	0.14	9.22	0.77
1100°C 60 min-2	0.10	9.54	0.54
1100°C 60 min-3	0.31	10.66	1.45
1200°C 10 min-1	0.07	9.63	0.36
1200°C 10 min-2	0.17	10.95	0.80
1200°C 10 min-3	0.10	11.20	0.45

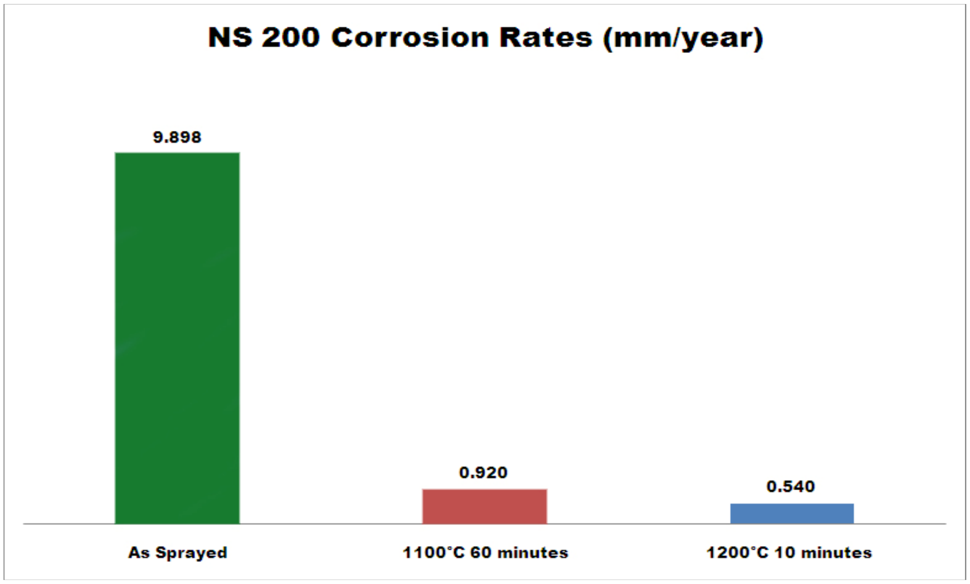

Figure 11: Average Corrosion rates for NS 200

Because C276 failed under all conditions (As sprayed and after heat treatments) it is obvious that Nicko-Shield 200 outperforms C276. On average, the corrosion rate of Nicko-Shield 200 was 9.90 mm/year for the as-sprayed condition, 0.92 mm/year after a heat treatment at 1100°C for 60 minutes, and 0.54 mm/year after a treatment at 1200°C for 10 minutes. Statistical analysis of the data was performed using Minitab statistical software. This analysis found that there was no statistically significant difference between the corrosion rates of the heat treated specimens, but that both heat treatments resulted in rates that were significantly lower than the as-sprayed specimens.

Discussion

Porosity

The porosity data collected in this experiment found that the area percent porosity both alloys in their as sprayed condition were not statistically different. In addition, the heat treatments performed on the alloys (1200°C for 10 min and 1100°C for 60 min) showed no

statistically significant difference from the as sprayed condition. These results are contrary to the original expectation that heat treatment would reduce the total porosity through a sintering like process. We believe that it is possible that the heat treatments performed this experiment simply were either not high enough temperature or were not held for a long enough time for diffusion to produce a measureable change in the porosity. However due to the fact that data was not able to be collected for the highest temperature heat treatment for a longer period of time than 10 minutes it is not possible to make any correlation between the porosity and the time parameter.

Another possible explanation for lack of measured change could be that there was actually a change in the porosity that was not reflected by the measurement technique used in this experiment. The technique used to measure porosity in this experiment provides a measurement of the total area percent porosity of the coating. This type of measurement provides no information about size, shape, number or distribution of the pores in the coating. It is possible that there was a change in the shape or number of pores that was in fact affected by the heat treatment but was not reflected in a measurement of total pore area.

Corrosion

Despite the fact that heat treatment of the TSC specimens produced no significant change in area percent porosity, the heat treated NS 200 specimens had significantly lower corrosion rates than the as-sprayed specimens. These findings do not support our original hypothesis: heat treatment will reduce porosity, which will in turn improve corrosion resistance. Since a decrease in percent porosity was not the cause of improved corrosion resistance, there must be another reason for the results we saw. We developed two possible explanations for the decreased corrosion rate of the heat treated specimens. The first was that heat treatment had some sort of effect on the size and distribution of pores in the coating, while the total area percent porosity

remained unchanged. The second, that during heat treatment, a reaction took place on the surface of the coating and produced a corrosion-resistant oxide layer.

High amounts of porosity are detrimental to a coating's corrosion resistance because the increased surface area allows more corrosive media to contact the coating's surface. Surface-connected pores thus pose more of a threat than pores deeper in the coating. Several small pores have more surface area than a single large pore of the same volume. Thus it is possible that while total area percent porosity remained unchanged after heat treatment, the size and distribution of the pores changed. With the software we had at our disposal, it was impossible to characterize the distribution and size of pores, so we re-examined the SEM coating images to look for qualitative evidence. SEM images of a typical specimen of NS 200 before and after heat treatment is shown below in Figure 12.

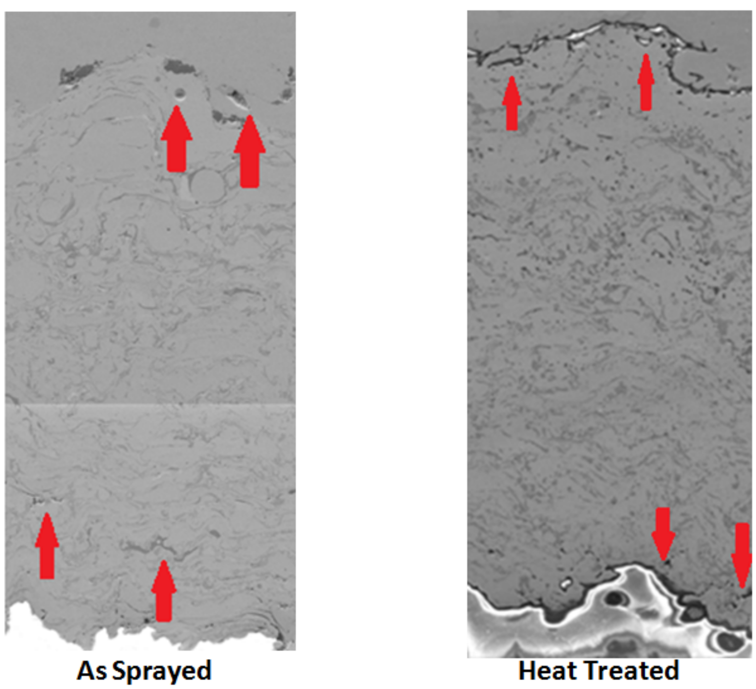

Figure 12: The distribution of pores remained relatively unchanged after heat treatment.

As shown, for the as-sprayed specimen, most of the porosity was found near the surface of the coating. The distribution did not change appreciably after heat treatment. We were not able to

detect a significant change in average pore size either, though more advanced software would be needed to confirm this. Thus, we can rule out this theory.

Our second theory is based on the suspicion that an oxide layer formed on the coating surface during heat treatment. Since we do not know the exact composition of our coatings, it is difficult to say what the composition of the oxide could be. The heat treatments were supposed to be carried out in an inert environment, preventing any oxide-forming reactions, but examination of the coating surfaces suggests perhaps the heat treatments were not performed as specified. Specimens of NS 200 in the as-sprayed condition and after heat treatments of 1100° for 60 minutes and 1200°C for 10 minutes are shown below in Figure 13.

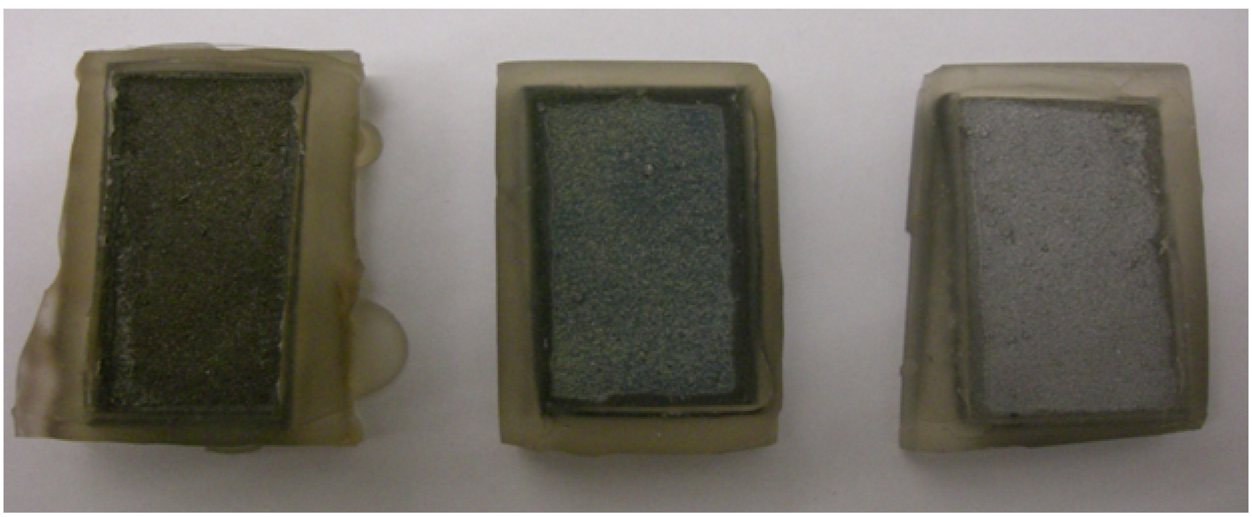

Figure 13: As-sprayed, 1100°C for 60 minutes, and 1200°C for 10 minutes specimens were all slightly different in color.

Each specimen is a distinctly different color: the as-sprayed specimen was dark grey, and the heat treated specimens were grayish-blue and light grey. The difference in appearance between these specimens leads us to believe that a reaction may have occurred during heat treatment that resulted in the formation of a new surface layer. The different colors could be due to differences

in thickness of the newly formed layer. Further characterization studies would have to be performed on the coating to verify this theory.

Conclusions

Analysis of the porosity and corrosion data collected in this study has led us to the following conclusions:

- C276 failed the corrosion test under all conditions
- Heat Treatment did not significantly affect area percent porosity
- Corrosion behavior of NS 200 was improved with heat treatment
- Corrosion behavior was not affected by porosity, it may have been improved by the formation of a oxide layer during heat treatment

Scoperta will be satisfied to hear that their alloy outperforms a conventional TSC alloy. Further studies are needed to determine if a reduction in porosity does in fact improve corrosion resistance. Using longer, hotter heat treatments, or more detailed analysis of the size, number and distribution of the porosity may give us better results. Further analysis of the heat-treated TSC surface would be needed to characterize the newly-formed surface layer and determine if this layer was the cause of the improved corrosion behavior.

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