

Morphology and Roughness of Al_2O_3 on Ferritic Stainless Steel Foils for Diesel Catalytic Converter Applications

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Approval page

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Abstract

The improved thermal properties of metal-substrate catalytic converters enable effective catalysis sooner after ignition than traditional ceramic monoliths, resulting in less harmful emissions. ACAT Global, a manufacturer of stainless steel catalytic converters, uses ferritic stainless steel substrates for low temperature catalytic converters for diesel automobiles. Production begins with an oxidizing heat treatment to increase surface roughness. The success of the second production step, the application of catalyst-containing ceramic slurry (washcoat), depends on the surface roughness that results from oxidation. Formation of the primary oxide of interest, alumina (Al_2O_3), is controlled by the stainless steel foil composition, oxidation temperature, and oxidation time. ACAT intends to use a specific foil, so composition will be constant. Temperature will also be constant; 900°C is most likely to result in growth of rough alumina since diffusion of aluminum to the surface is faster than the diffusion of oxygen into the material at that temperature. This project focuses on the effect of the last parameter, treatment time, on oxide morphology and roughness. Samples were removed at hour intervals of a six hour 900°C hold. Qualitative assessment of surface morphology was performed using SEM, revealing that “rough” oxide resulted from treatment times exceeding three hours at temperature. Additionally, SEM images revealed an increase in the frequency and magnitude of surface features with increasing treatment time and a decrease in the frequency and size of dark regions of smooth oxide. Profilometry was used to collect R_a roughness values of the rough oxide samples. Average profile roughness increased from 217 nm for the untreated foil to a maximum value of 564 nm for the five hour sample. The decrease in R_a from five to six hour samples was unexpected and could be a consequence of limited vertical resolution of the profilometer settings. Oxidation times of four, five, and six hours appear to have the rough morphology preferred for washcoat adhesion. Specific interactions of the washcoat with features of various heights and lateral spacings should be assessed to determine ideal oxidation time.

Keywords: Materials Engineering, Catalytic Converter, Oxidation, Ferritic Stainless Steel, Alumina, Profilometry

Introduction

Purpose

The objective of this project was to coordinate with ACAT Global (Charlevoix, MI), a manufacturer of stainless steel-substrate catalytic converters, to improve processing of stainless steel foils for use in catalytic converters. The study focused on a particular processing step for the converters: foil oxidation, which is performed to grow an alumina oxide with sufficient roughness and coverage for good adhesion of a catalyst-containing washcoat. Without oxidation, the surface of the stainless steel foil is smooth and the washcoat will not adhere well. The oxidation of ferritic (Fe-Cr-Al) stainless steel foils used for metal substrate catalytic converters for diesel automobiles was characterized. Of the factors that affect oxide formation, oxidation time was of particular interest to maximize part throughput and minimize part failure. Since a rough oxide is essential for washcoat adhesion, foils were characterized qualitatively to identify oxide morphology as “rough” or “smooth”, and quantitatively by R_a profile roughness values.

Catalytic Converter History

The invention of the catalytic converter resulted from Los Angeles’s Federal Motor Vehicle Act, enacted in 1960, which required that research be conducted on air pollution from automobiles. The legislation was preceded by recognition of, and concern surrounding, smog in Los Angeles in the 1940s. Episodes of smog were marked by reduced visibility, eye and respiratory discomfort, nausea, and vomiting and was attributed to the increase of industrialization and personal automobile use in the city. To reduce the presence of smog and the unfortunate side effects associated with it, technologies were investigated to address the effects of automobile emissions, an apparent contributor. Despite the development of several technologies, including catalytic converters, implementation of emission-reducing devices was not required until California adopted the first hydrocarbon (HC) and carbon monoxide (CO) emission standards in the United States in 1966.¹

Concern about air quality increased as the adverse impacts of pollution on human and environmental health were identified. In response to increasing concern and regulation, the first catalytic converter was manufactured in 1975.¹ These “two-way” converters used the heat of the engine exhaust to oxidize

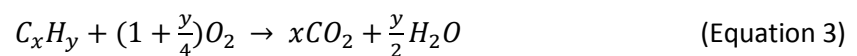
CO and HCs into water and carbon dioxide (CO₂).² Modern three-way converters developed later and treated nitrous oxides (NO_x) in addition to CO and HCs.²

Automobile emission regulations, and technology to meet regulation requirements, continue to develop currently, and metal substrate catalytic converters offer improved conversion relative to traditional ceramic technologies. Thermal and mechanical properties of metal-based converters promise systems that can meet increasingly stringent requirements as well as minimize harmful pollutants by more effectively converting combustion byproducts into less harmful emissions.³

Catalytic Converter Function

Catalytic converters process the more damaging byproducts of engine combustion and convert them to less harmful substances. Gas and air react in combustion to power automobiles, with major byproducts of HCs, NO_x, and CO. Pollutant byproducts are converted by reaction at the catalytic converter surface into less harmful emissions of water vapor, nitrogen gas (N₂), and carbon dioxide (CO₂). Although CO₂ contributes to global warming, emissions are considered benign relative to pollutants which have more adverse effects on environmental and human health. NO_x contribute to smog, acid rain, and irritation of human mucus membranes; HCs contribute to smog; and CO is odorless, colorless, and poisonous.⁴

As the name suggests, catalytic converters use catalysts (platinum, palladium, and rhodium), which increase the rates of the oxidation and reduction reactions responsible for conversion (Equations 1-3) without being consumed.⁴ Conversion occurs at the catalytic converter surface and, as such, can be improved by increasing the surface area available for reactions. Despite the benefit of increased surface area, catalytic converter design must balance surface area with sufficient air flow through the converter and the converter size allowable within automotive design constraints.



Factors affecting conversion efficiency of catalytic converters include the air-to-fuel ratio of the combustion reaction, the catalytic converter operating temperature (affected by proximity to engine), and geometrical characteristics of the air flow channels (Figure 1).³

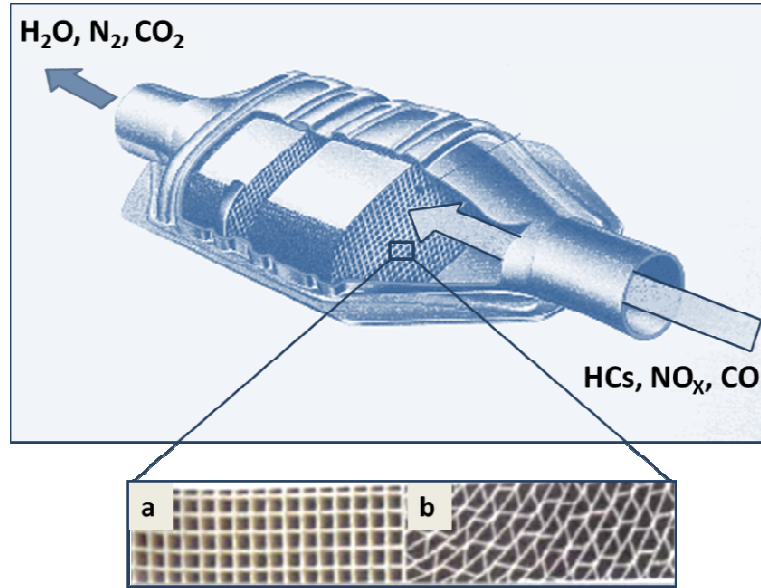


Figure 1: Schematic representation of combustion byproduct conversion to emissions after traveling through the catalytic converter channels; traditional ceramic converters with honeycomb channels (a) have less surface area than channel geometries achievable in metal substrate catalytic converters (b).

Metal and Ceramic Substrate Catalytic Converters

Catalytic converters were first made from ceramic substrates (monoliths) with an internal honeycomb structure to provide a large surface area for conversion without inhibiting air flow.³ Metallic monoliths were first considered as an alternative to ceramic converters in the 1990s. A metal substrate offers advantages over ceramics since the metal foil can be processed to thinner dimensions and produced with alternative geometrical cross-sections, both of which provide more surface area in the same volume while maintaining mechanical stability.⁵

Wall thickness and channel configuration of metal substrate monoliths increase surface area in the same part volume which provides more surface area for catalysis and improved air-flow through the channel, both of which enable more effective conversion of pollutants to less harmful emissions.³ A good indicator of relative air flow can be inferred from the frontal flow area of the monolith: in a 400 cell per inch ceramic monolith, 61% of the frontal area is open whereas 91% of metallic monolith frontal area is

open.³ Metal substrates are also desirable for their superior thermal shock resistance, which reduces the risk of part failure by thermally-induced cracking.³ The high thermal conductivity and low heat capacity of metallic monoliths relative to ceramics enable shorter light-off times.

Light-off time is the time required for the converter to reach the temperature at which conversion is most efficient. The highest percentage of unconverted pollutant emissions in ceramic converters occurs during the first minute of automobile operation; therefore, the reduced light-off times achievable with metallic monoliths result in better overall emission conversion by reaching efficient conversion sooner than ceramic converters. Shorter light-off times provide an advantage as increasingly stringent emission regulations exceed the capabilities of traditional ceramic monoliths.³

Catalytic converters of each substrate material have similar processing steps: the monolith is created, the monolith surface is prepared chemically and/or physically, and a catalyst-containing washcoat is applied. Good washcoat adhesion is critical to catalytic converter performance and can be a challenging aspect of the manufacturing process. For metal substrate converters, ferritic stainless steel alloys with Cr and Al content are pretreated (oxidized) to form a layer of aluminum oxide (alumina, Al_2O_3), which has both the physical morphology and chemical reactivity to promote washcoat adhesion.

Surface treatment to reduce processing time and improve washcoat adhesion is the focus of this project. The following sections provide background specific to the parts and processes encompassed by the scope of the project, including desired oxide characteristics for good adhesion and processing specific to ACAT Global.

Overview of ACAT Processing

ACAT's manufacturing process has three main steps: (1) monolith assembly, (2) oxidation, and (3) application of catalyst-containing washcoat. (1) Foils received from the manufacturer undergo mechanical deformation from flat to corrugated foil which is then wound into the monolith configuration and welded at metal contact points. Proper component function relies heavily on (2) the chemical and physical preparation of the substrate surface for (3) the application of the catalyst-containing washcoat. A schematic representation of the cross section of a processed catalytic converter foil is depicted in (Figure 2). The second step is crucial to the effectiveness of subsequent processing

steps and component operation, and is time intensive. As such, this project focused on oxidation of stainless steel to reduce processing time without compromising washcoat adhesion.

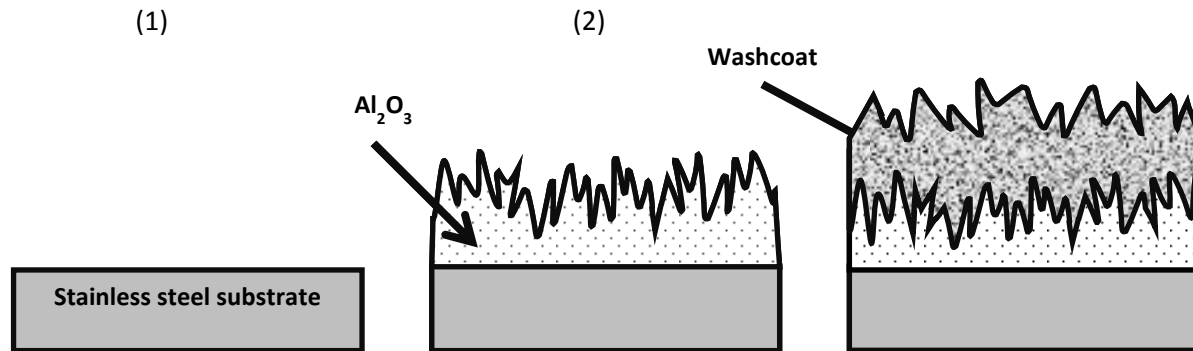


Figure 2: Schematic representation of the foil surface after corrugation and monolith assembly (1), surface preparation by oxidation (2), and washcoat application (3).

Diesel engines have a lower operating temperature than gasoline engines. Consequently foil compositions specific to engine type are used to minimize the cost required to meet performance requirements.³ The foil of interest for this project was for diesel applications, for which ACAT uses Diesel Foil (DSF) with specific composition (Table I).

Table I – Nominal Composition of DSF in Weight Percent

Cr: 14.7-16.4	Al: 5.0-6.0	C: <0.08	Mn: <0.8	Si: <0.8
S: <0.01	Fe: Balance with trace amount of other residual minor elements			

Oxidation of DSF

Stainless steels generally contain higher concentrations of chromium and aluminum than carbon steels and sometimes contain rare earth elements (REEs), such as yttrium or lanthanum, which alter initial and long-term oxidation.⁶ The “stainless” property associated with stainless steel results from the formation of a passive chromium-rich oxide surface film that is generally continuous, nonporous, and insoluble in the presence of oxygen at low temperatures.⁷ At higher temperatures, more notable oxides form with different composition and morphology than is seen at room temperature. So, while stainless steel does not appreciably oxidize at room temperature, significant oxidation will occur at elevated temperatures.

Environment, alloy composition, temperature, and time affect the composition and morphology of oxides that form at elevated temperatures and can be used to tailor oxide properties to suit a specific

application.⁸ Metal substrate catalytic converter processing relies on the formation of alumina with rough morphology for effective application of the washcoat. Additionally, alumina has the thermal stability required for elevated operating temperature catalytic converters.⁹ Despite excellent performance in the application, achieving alumina with desirable morphology and sufficient roughness can be challenging. Increased surface roughness benefits washcoat adhesion and can be achieved by balancing the parameters responsible for oxide composition and morphology.

Previous research by General Motors/Delphi suggests a correlation between α -alumina, which has a rough morphology (Figure 3), and successful washcoat adhesion.¹⁰ The qualification of rough is attributed to alumina with high aspect ratio plate or whisker morphology features with sharp edges. Full coverage of the surface with a rough oxide is also preferable, as the washcoat is not expected to adhere well to smooth oxide areas.

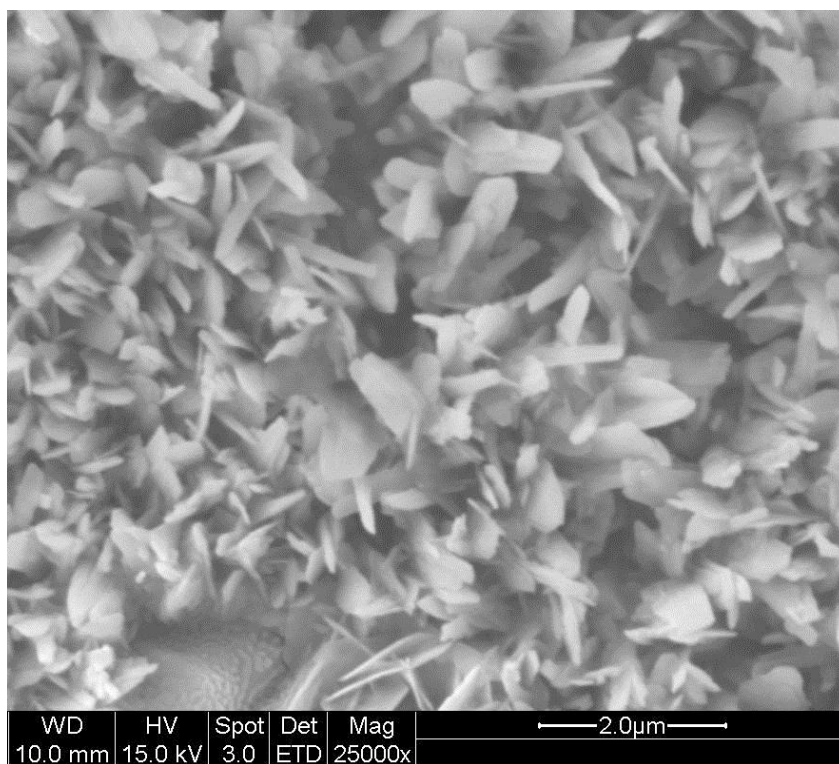


Figure 3: SEM micrograph of a rough oxide formed during a six hour hold at 899°C expected to have good washcoat adhesion.

Environment, alloy composition, temperature, and time were considered in attempting to improve washcoat adhesion. Environment is fixed based on ACAT furnaces, which are open to the ambient atmosphere. Alloy composition will also be fixed for DSF, which contains Cr and Al, but no REEs, which

stabilize oxidation during catalytic converter operation and have an effect on initial oxidation.⁶ Oxidation temperature will also be fixed; near 900°C the slow diffusion of oxygen into the material relative to the diffusion of Al to the surface promotes the formation of alumina at the solid-gas interface. Conversely, near 1200°C the diffusion rates of oxygen and Al are much closer, resulting in internal alumina formation which does not provide the morphology of interest for catalytic converter processing.¹¹ According to the constraints on other parameters, time was varied with an interest in how oxidation progressed over time. Additionally, varying oxidation time provided information of the minimum time required to form rough alumina.

Realistic Constraints¹²

The primary objective of this project was to modify the oxidation treatment of metal substrate catalytic converters to improve part throughput and maximize part performance. In addition to the scientific exploration and the contribution to ACAT Global's processing, this project will have broader impacts and must take place within realistic constraints applicable to any engineering project. Improved processing and performance of catalytic converters will have a particular effect toward reducing the impact of automobiles on human health and safety and the environment.

Health and Safety

As previously mentioned, there are human health and environmental quality concerns associated with engine combustion byproducts. Accordingly, there is interest in the continued use and development of catalytic converter systems with improved conversion efficiency. Some of the major pollutants that challenge human health are CO, ozone, sulfur dioxide, and NO₂. CO interferes with the effective delivery of oxygen to vital organ systems. Young children and individuals with heart disease are especially susceptible to the effects of CO exposure, but even healthy individuals may experience minor symptoms in the form of headaches and fatigue. HCs and NO_x pollutants combine in the atmosphere in the presence of light to form ozone. Exposure to ozone can cause inflammation of lung tissue and breathing passages, decreasing the ability of one's lungs to work effectively. Sulfur dioxide, a byproduct specific to the combustion reaction of diesel fuel, and NO₂ constrict airways and are particularly harmful for children and people with asthma.¹³

Pollutants put susceptible individuals at risk and reduce the well-being of otherwise healthy individuals. Shortening the light-off time of catalytic converters and increasing the area upon which catalysis and conversion can occur both aid in improving efficiency and can be achieved with the development and implementation of metal substrate catalytic converters. This improved performance can be attributed to the improved thermal properties and channel geometries achievable with metal substrates increase the conversion efficiency and reduce emission of harmful pollutants during vehicle operation.^{13, 14}

Environmental Impact

In addition to the reduced quantity of harmful emissions resulting from catalytic converter use, metal substrate catalytic converters provide other environmental benefits. A particular value is in the increased recyclability of metal substrate converters relative to their ceramic counterpart. Recycling of ceramic monoliths is limited to the extraction of platinum, palladium, and rhodium. Alternatively, there is a procedure for the recovery of both steel and precious metals from metal substrate catalytic converters.¹⁴ Since metals are more recyclable than ceramics, greater percentage of materials in metal substrate converters can be recycled, yielding a reduction in the lifecycle impact associated with catalytic converters. Increased recyclability reduces the amount of waste sent to landfills and the energy required for raw material generation. For metal substrate converters, the catalyst containing ceramic layer can be mechanically removed from the substrate, so less material has to be processed for precious metal recovery. The improved recovery of catalytic materials during recycling eases some of the need for mining them, an environmentally harmful process.¹⁴

Methods and Materials

Project Plan

The efficient performance of metal substrate catalytic converters relative to their ceramic counterpart was the impetus toward investigating alumina growth as a means to improve their processing. Since temperature and composition conditions are fixed, samples were treated to an increasing number of hours at an oxidation temperature of 899°C. The resulting alumina formation was evaluated qualitatively by scanning electron microscopy (SEM) and quantitatively by R_a roughness values from profilometry.

Oxidizing Heat Treatment

As mentioned above, the diffusion of oxygen into the stainless steel alloy is slow relative to the diffusion of aluminum to the surface near 900°C, which is ideal for forming alumina on the substrate surface. Accordingly, samples were oxidized at 899°C (1650°F). A CM 1710 FL furnace with high temperature, ramping, and reliable temperature regulation capabilities was used for oxidizing heat treatments (Figure 4).



Figure 4: Programmable CM 1710 FL furnace capable of heat treatment profile, high temperature, and temperature control required for the study.

An approximately eight hour treatment was performed (Table II). Ramping to temperature was designed according to ACAT furnace capabilities and had two regions, one in which temperature increased from room temperature to 538°C (1000°F) in 50 minutes and a second in which temperature

increased at a slower rate to the oxidation temperature of 899°C (1650°F) in 65 minutes. The treatment profile (Figure 5) shows the temperature and time of ramps and holds. Also included are the times at which samples were removed. In order to observe the progression of alumina formation over time, samples were removed after ramping and after 1, 2, 3, 4, 5, and 6 hours at the oxidation temperature. An untreated sample was also evaluated for reference.

Table II – Oxidation Treatment Ramping and Hold Conditions

Cycle Step	Temperature (°C)	Temperature (°F)	Time (min)
Ramp 1	0 - 537.8	0 - 1000	50
Ramp 2	537.8 - 898.9	1000 - 1640	65
Hold	898.9	1640	360

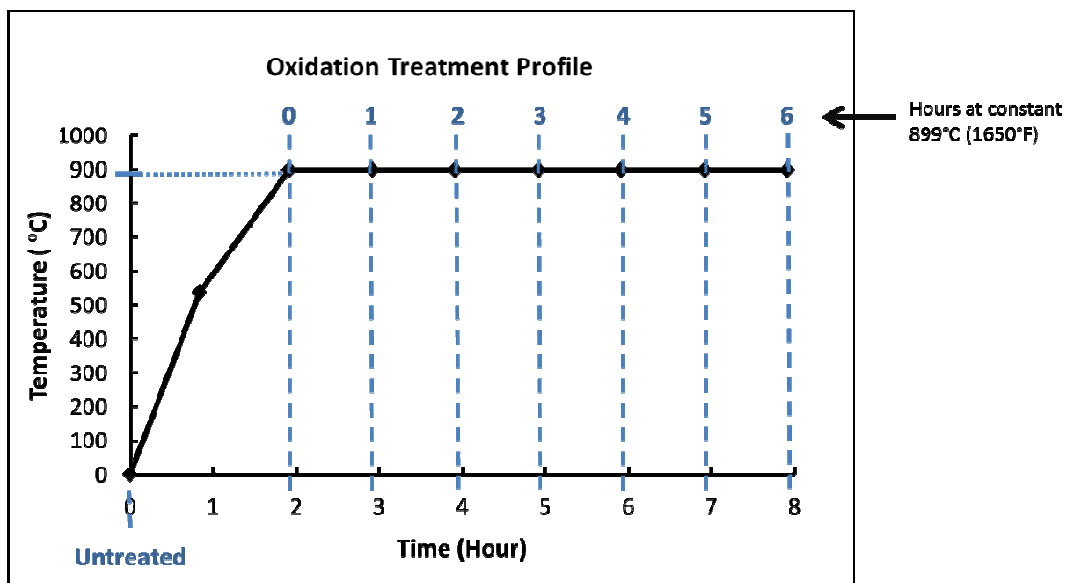


Figure 5: Time and temperature profile of oxidation showing the points at which samples were removed: untreated and at 0, 1, 2, 3, 4, 5, and 6 hours at 899°C (1650°F).

Sample Preparation

Upon receipt from ACAT, foil samples were corrugated, but not yet wound or welded into monoliths. It was anticipated that corrugation added cold work that might affect oxidation since dislocation content and other high energy areas affect diffusion.⁸ The foil was cut into samples approximately 4 cm long and 1 cm wide. For ease of microscopy, one foil for each sampling condition was flattened by mechanical

pressing (Figure 6). Flattening enabled the resolution and magnification required for morphological characterization using SEM.



Figure 6: Corrugated (a) and flattened (b) foil samples.

Although flattening resulted in additional cold work to the foil, the areas of interest, the flat planes between peaks and valleys of corrugation (Figure 7), were assumed to experience only minor deformation. The flat planes are most relevant since the some of the peaks will be welded to adjacent foils in constructed monoliths and represent the majority of the monolith surface.

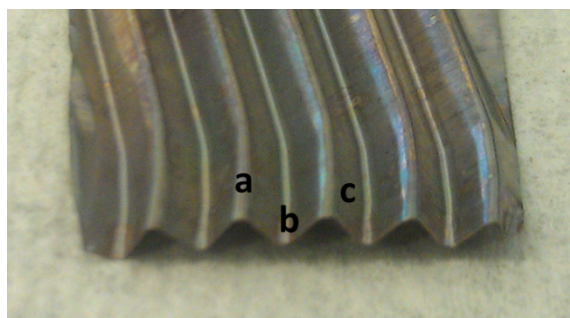


Figure 7: Side view of corrugated foil with peaks (a), valleys (b), and flat planes (c) anticipated to have different oxidation behavior.

To evaluate the possible effect of flattening on alumina formation and for quantitative R_a measurements using profilometry, corrugated samples were also treated. Before oxidation, samples were rinsed with ethanol, in accordance with ACAT practices, to remove any contaminants from corrugation, flattening, and handling.

SEM uses electrons to create images; accordingly, imaging electrically conductive samples enable better resolution at high magnifications than non-conductive samples. The alumina on foil samples was electrically insulating, so gold was deposited onto the surface using a timed sputtering process to

increase sample conductivity. Sputtering took place in a chamber purged with nitrogen and at 100 mTorr. A 300 Angstrom layer of gold was deposited in 300 seconds of sputtering and greatly improved the resolution achievable in SEM imaging and, consequently, the ability to qualitatively evaluate alumina morphology.

Characterization

Alumina Morphology

A FEI Quanta 200 SEM was used to collect images of alumina morphology for qualitative evaluation. Images were used to categorize alumina on each sample as rough or smooth depending on feature aspect ratio and morphology. Rough oxide was characterized by sharp-edged plates and whiskers whereas smooth oxides were shorter and spherical. Coverage with rough oxide was also considered for evaluation of overall roughness. Resolution at high magnification was greatly improved by flattening and sputtering samples for microscopy.

Sample Roughness

Several types of equipment were considered for quantitative roughness measurements including ellipsometry, optical interferometry, atomic force microscopy (AFM), and profilometry.^{10, 15} Based on equipment access, neither ellipsometry nor optical interferometry was possible for this project. AFM would have been beneficial as it can provide three dimensional surface representation as well as numerical profile roughness values. Due to the small area upon which AFM measurements are based and limited availability, AFM was not a practical method for gathering measurements representative of the whole surface. Profilometry was a more feasible alternative for quantitative roughness measurements. In addition to increased availability and shorter scan time, profilometer measurements compiled data from millimeter length scans, as opposed to the micrometer limitations of the AFM, providing R_a profile roughness values more representative of the whole sample. A fortunate consequence of short scan times was the opportunity to perform more scans than would be possible using AFM; data from twenty scans was used to evaluate R_a for untreated and 4, 5, and 6 hour hold samples.

Profilometer scans were performed with a scan speed of 0.03 mm/s using 1 mg of force over 1 mm scan lengths on flat planes of the corrugated samples. Data was linearized to accommodate macro-scale

vertical variation that resulted from the slope of the plane depending on sample orientation relative to the scanning stylus. Quantitative output considered for comparison was R_a , the arithmetic mean of vertical deviations along the scanned profile.

Results and Discussion

Alumina Morphology

SEM micrographs were gathered for flattened and sputtered samples of untreated foil and foil oxidized at 899°C (1650°F) for 3, 4, 5, and 6 hours. Oxidation was evident on the 3 hour hold sample, but had a spherical, nodule morphology (Figure 8a). Rough oxide was apparent on 4, 5, and 6 hour hold samples, identified by sharp-edged plates and blades (Figure 8b, c, and d). The same micrographs showed a dark region in the 5 hour sample; upon further investigation, it was found that this dark area contained shorter and smoother oxide. Similar regions were evident on the 4, 5, and even 6 hour samples (Figure 9). Additionally, differences were observed between regions of rough oxide; coarse and fine variations of rough oxide were present on all treated samples (Figure 10).

Size and frequency of dark regions and evidence of rough oxide were considered in evaluating alumina formation by examining rough oxide coverage of large areas at low magnification. Shades on the micrographs corresponded to the relative height of features on each sample surface; short features, such as regions of short and smooth oxide, appeared dark while more developed, rougher oxide had lighter shading. The size and frequency of smooth oxide regions decreased as oxidation time increased (Figure 11).

Untreated samples showed an absence of significant surface features with significant vertical deviation, as was expected since only the room temperature chromium oxide would be present before oxidation. There were some low aspect ratio surface features perpendicular to corrugation, which appeared to be a result of cold work in the direction of rolling during corrugation (Figure 12). Similar lateral directionality was apparent in alumina formation on treated samples (Figure 13), indicating that the pretreatment surface condition affected oxidation.

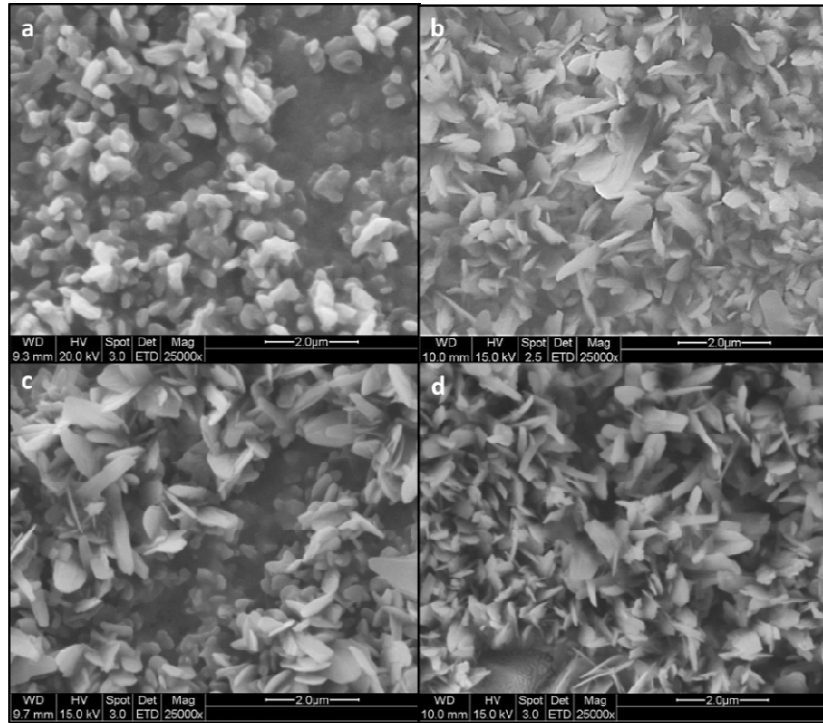


Figure 8: Smooth alumina on the 3 hour hold sample (a) and rough oxide on samples treated for 4 (b), 5 (c), and 6 (d) hours.

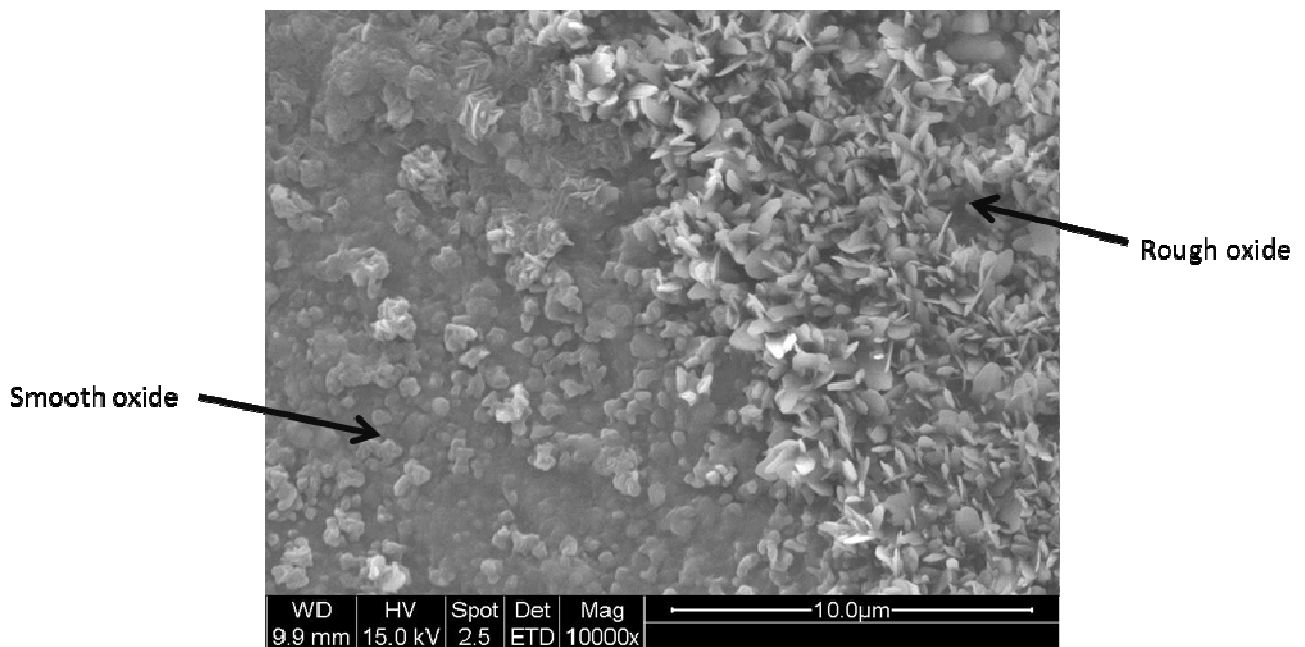


Figure 9: The 6 hour hold sample, as all treated samples, had regions of smooth oxide (left), which appeared dark relative to rough oxide (right).

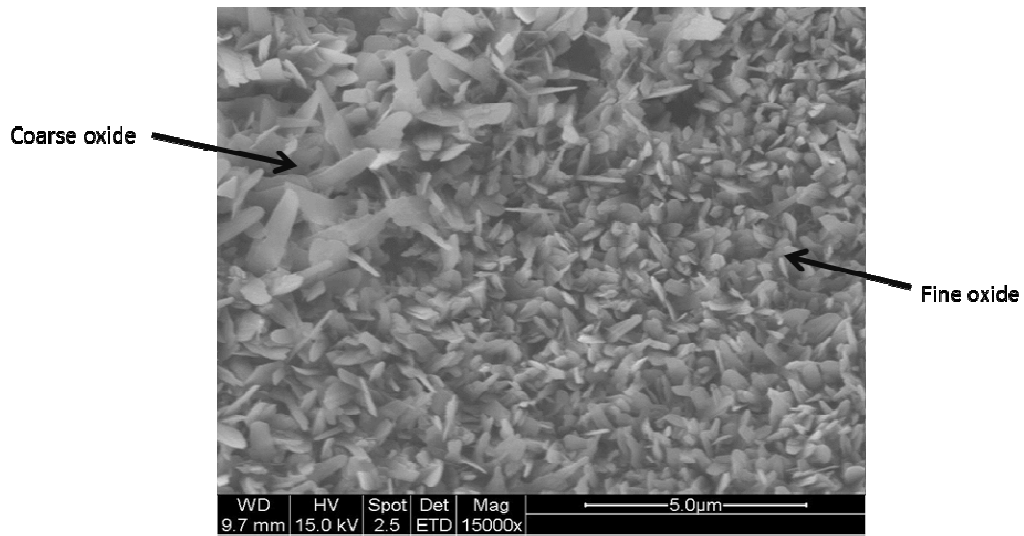


Figure 10: Areas of coarse (upper-left) and fine (lower-right) rough oxide on the 5 hour sample.

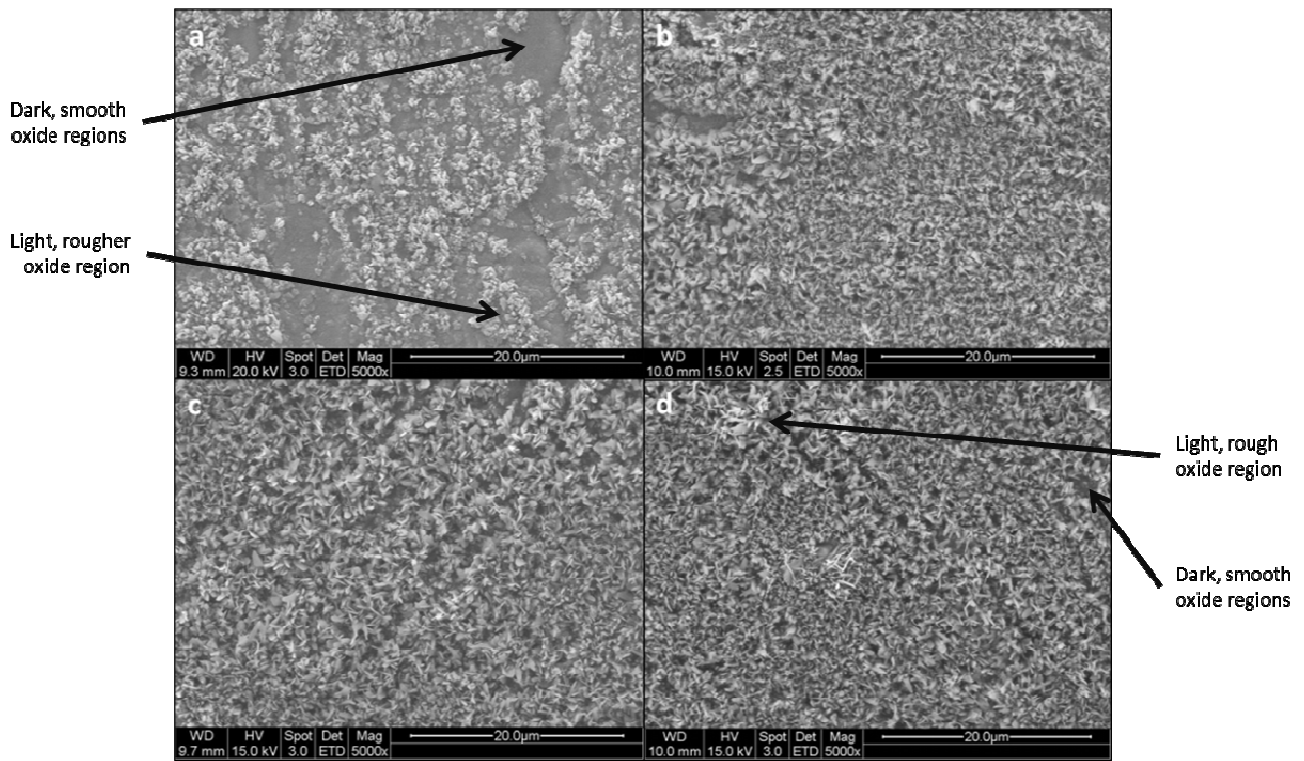


Figure 11: All oxidized samples had regions void of rough oxide, indicated by dark regions of smooth oxide. The 3 hour sample (a) contained more smooth oxide than rough oxide; size and frequency of dark regions decreased as oxidation time increased as can be seen between the 3 (a), 4 (b), 5 (c), and 6 (d) hour samples.

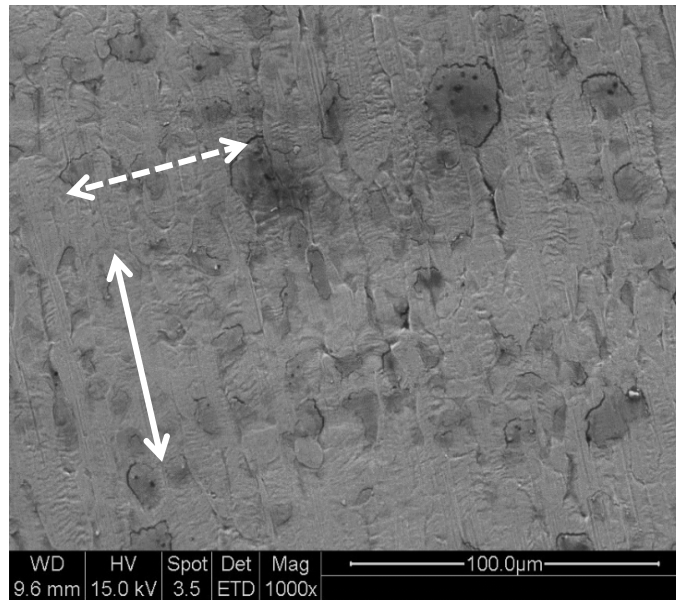


Figure 12: Untreated sample surface showing lateral features in the direction of rolling during corrugation (solid arrow) that occur perpendicular to corrugation (dashed arrow).

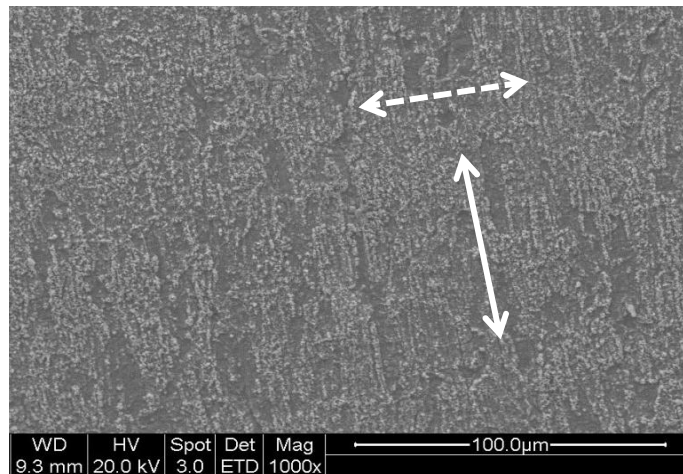


Figure 13: 3 hour sample surface showing lateral features in the direction of rolling during corrugation (solid arrow) that occur perpendicular to corrugation (dashed arrow) and reflect features on the untreated sample surface.

The oxidation along discontinuities in the initial sample surface implies a correlation between oxide formation and high energy areas at which diffusion is generally preferred.⁸ For this study, an attempt to increase surface roughness, the increased vertical formation of alumina appears to benefit processing. At longer oxidation times or extreme lateral surface variation before oxidation, however, there is a risk of excessive oxidation with potential results including, but not limited to, reduced mechanical stability of oxide platelets, reduced adhesion of the oxide to the metal substrate, or a combination of the two.

Sample Roughness

Profilometry scans provided quantitative assessment of how roughness varied with oxidation time. Corrugation ridges were used to align the stylus along a scan path with minimal macro-scale displacement due to the sample slope. The flat planes, rather than the peaks or valleys, were scanned (Figure 7). An untreated sample was used as a control and the samples that exhibited qualitatively rough oxide in SEM analysis were compared by R_a roughness measurements (Table III).

Table III – Average R_a Roughness Measurements

Sample	Mean R_a (nm)
Untreated	216.68
4 hour	366.47
5 hour	564.36
6 hour	402.19

As expected, R_a increased from untreated to treated samples. There was a surprising difference between the small R_a increase from untreated to 4 hours at oxidation temperature and the relatively large increase from 4 to 5 hour samples. The most unexpected result, however, was the reduction in R_a between 5 and 6 hour hold samples. A Tukey pairwise comparison of a one-way ANOVA of R_a data produced the plot in Figure 14 and confirmed a statistically significant difference in roughness between all samples except for the 4 and 6 hour samples.

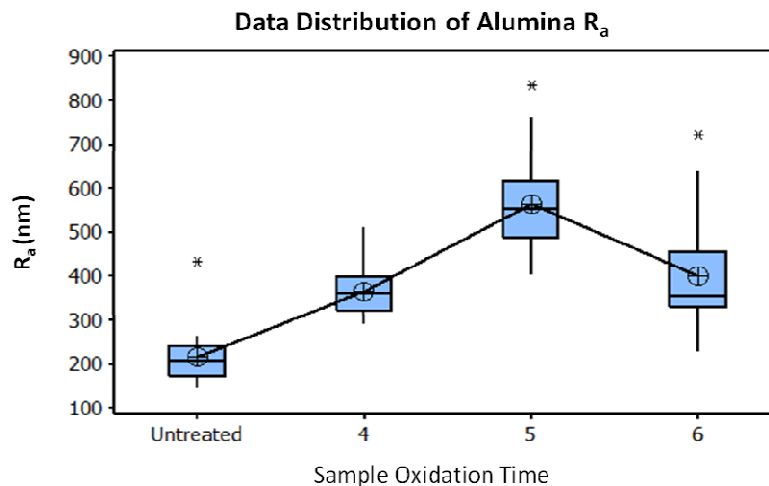


Figure 14: R_a values generally increased with oxidation time, except for between the 5 and 6 hour samples; there were statistically significant differences between all samples except for the 4 and 6 hour samples.

The decrease in R_a from 5 to 6 hours of oxidation was unexpected and prompted reflection on morphology images to identify a source of the observed trend. One possible cause of the unanticipated variation was the change in size and frequency of dark, smooth oxide regions. Specifically, there was a possibility that the decrease in these regions from 4 to 5 hours resulted in increased vertical deviation over the scan profile length. Between the 5 and 6 hour samples, however, the further decrease in size and occurrence of these regions reduced the average vertical deviation because of the infill of these regions with rough alumina.

While the size and presence of dark regions between samples may have affected roughness measurements, a more likely contributor was the limited ability of the profilometer at the scan settings used. Provided the scan speed used for measurements as well as the orientation of the alumina plates having some lateral, rather than purely vertical, dimension, the stylus was likely unable to detect the full vertical depth between plates. A simplified schematic of the possible deviation between the sample profile and the profile detected for roughness calculations is provided in Figure 15.

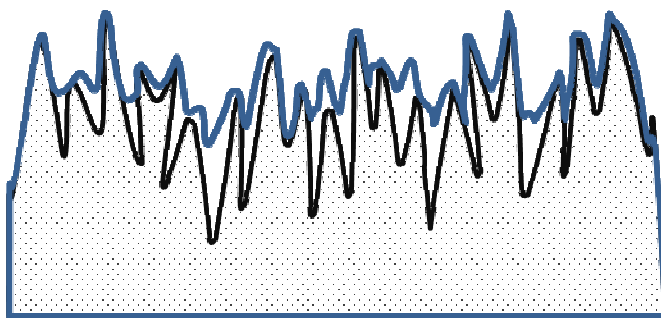


Figure 15: Simple schematic of the deviation between the oxide profile (black) and the detectable vertical displacement as measured with the profilometer under previously stated scan conditions (blue).

Upon evaluation of profilometry results, the most valuable conclusion that can be drawn from the results is the need for a better method of quantifying roughness. Additionally, a method that provides surface roughness (S_a) rather than profile roughness (R_a), such as optical interferometry, would provide more accurate information.

Conclusions

1. The sample treated oxidized at 899°C (1650°F) for 3 hours had only smooth oxide, which is not expected to have good washcoat adhesion, indicating that oxidation times greater than three hours should be considered.
2. Oxide formation on the 4, 5, and 6, hour samples were all categorized as rough overall, however, each contained regions of smooth alumina as well as a combination of coarse and fine rough oxide. The frequency and size of smooth regions decreased with increasing oxidation treatment time, indicating an increase in surface coverage with rough oxide with increasing treatment times.
3. Although profilometry data indicated a maximum profile roughness at 5 hours, an improved method of quantitative analysis is required for more accurate characterization.
4. While qualitative and quantitative characterization of the samples provided can aid in improving processing for stainless steel substrate catalytic converters, the specific interaction of the oxide with the washcoat must be investigated before an oxidation time can be recommended.

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