Catalytic Regeneration of Electroless Deposited Nickel on Silicon Carbide Ceramic Foam Diesel Particulate Filters

Hunter Starring[1], Patrick F. Mensah[2], Stephen Akwaboa[2], Lulit Affin[2]

[1]Department of Biological Engineering, Louisiana State University, Baton Rouge, Louisiana, USA
[2]Department of Mechanical Engineering, Southern University and A&M College, Baton Rouge, Louisiana, USA

LA-SiGMA
Louisiana Alliance for Simulation-Guided Materials Applications

NSF

Abstract

Diesel Particulate Filters (DPFs) are becoming a more effective solution to meet the stricter regulations being placed on diesel vehicles from the Environmental Protection Agency (EPA). However, DPFs are costly and a method of catalyzing the regeneration of these filters using metals is the focus of much research. We developed a novel method of Electroless Nickel Deposition onto the surface of Silicon Carbide (SiC) Ceramic Foam DPFs to harness the catalyzing characteristics of Nickel in the exhaust of diesel engines to develop a continuously regenerating filter. The characterization of Nickel (Ni) on the SiC DPFs surface was confirmed using SEM/EDX. These filters were then tested in the exhaust of a 1-cylinder diesel engine using pure diesel fuel while chemical and differential pressure data was collected. In turn, we can confirm that SiC DPFs are sufficient in reducing particulate matter in diesel exhaust. However, only one of the two products of catalytic regeneration (NO2 and CO2) increased for the Ni Plated SiC filters.

Introduction

With the Environmental Protection Agency (EPA) placing stricter regulations on the emissions of light and heavy-duty vehicles, a large field of research is devoted to meet these regulations in a cost effective manner. The common solution to these regulations are Diesel Particulate Filters (DPFs). DPFs are used to collect the particulates, also referred to as soot, in the exhaust of diesel engines. These filters come in all different sizes and shapes. However, the filter with the most promise is a Silicon carbide (SiC) ceramic foam filter. This allows for the most surface area for adsorption of the exhaust particulates. However, these filters clog after some time and need to either be replaced or regenerated. These filters are expensive and the current process of regeneration is not energy efficient. Therefore, regeneration of DPFs is the focus of our research.

One aspect of meeting the EPAs regulations is to transfer vehicles to a more environmentally friendly fuel such as biodiesel. According to *Kosgei, et. Al.* one of the characteristics of biodiesel fuels is as the percentage of biodiesel increases, the Nitrous Oxide (NOx) concentrations increase. NOx consists of Nitrogen monoxide (NO) and Nitrogen dioxide (NO_2). According to *Jacquot et. al.*, NO_2 is very energetic and has the best oxidizing characteristics of all the chemicals present in biodiesel exhaust. Therefore, our task is to determine the best metal coating on a Silicon carbide ceramic foam filter that catalyzes the oxidation of soot by NO_2 present in biodiesel exhaust to create a continuously regenerating trap. However, first the efficiency of these filters must be tested using pure diesel fuel.

This catalytic regeneration (oxidation) can be seen below.

$$1.\ NOx + O_2 \rightarrow NO_2$$

$$2.\ NO_2 + C \rightarrow CO + NO$$

$$3.\ CO + NO + O_2 \rightarrow NO_2 + CO_2$$

Processes 1 and 3 occur spontaneously in a diesel engines exhaust, however process 2 does not. Therefore, process 2 is the reaction that must be catalyzed using a metal coating. To determine the success of the catalytic regeneration of the metal coating, a spike in NO_2 and CO_2 concentrations with a steady differential pressure value should be seen.

SOUTHERN UNIVERSITY

Electroless Deposition of Nickel

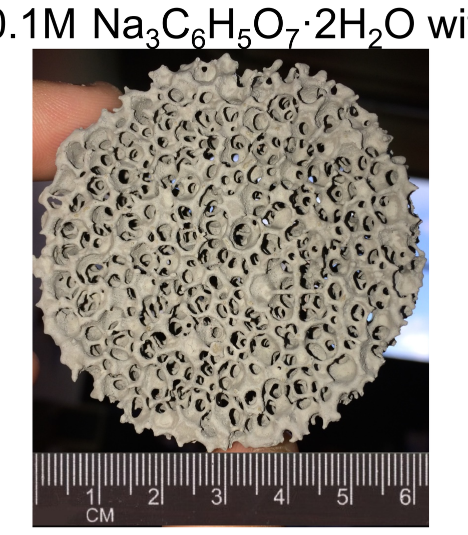
Electroless Deposition is a well established method to obtain a thin metallic film on an insulating substrate in a cost effective manner. However, this method is not widely used to coat metals onto DPFs. Therefore, we developed a novel method of Electroless Deposition of Nickel onto SiC ceramic foam DPFs. The SiC filter was pretreated and plated.

Pretreatment:

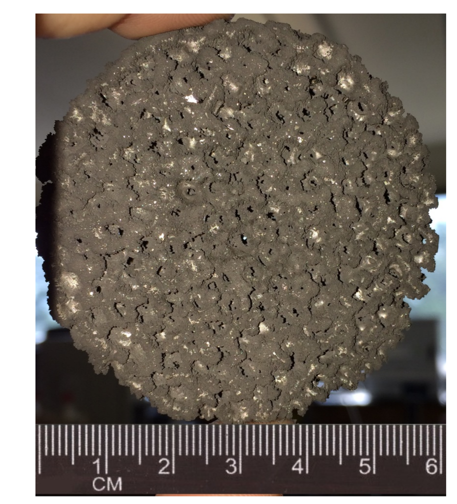
1) Acetone cleaning while sonicated. Etched in 20% Nitric Acid solution while sonicated, **2)** Tin sensitized in 0.04M $SnCl_2$, **3)** 0.25M HCl solution while sonicated, **4)** Palladium activated in 1.7×10^{-3}M $PdCl_2$, 0.157 M HCl solution while sonicated.

Plating:

1) Mechanically stirred in 0.1M $NiSO_4 \cdot 6H_2O$, 0.25M $NaH_2PO_2 \cdot H_2O$, 0.1M $Na_3C_6H_5O_7 \cdot 2H_2O$ with a pH between 9-9.5

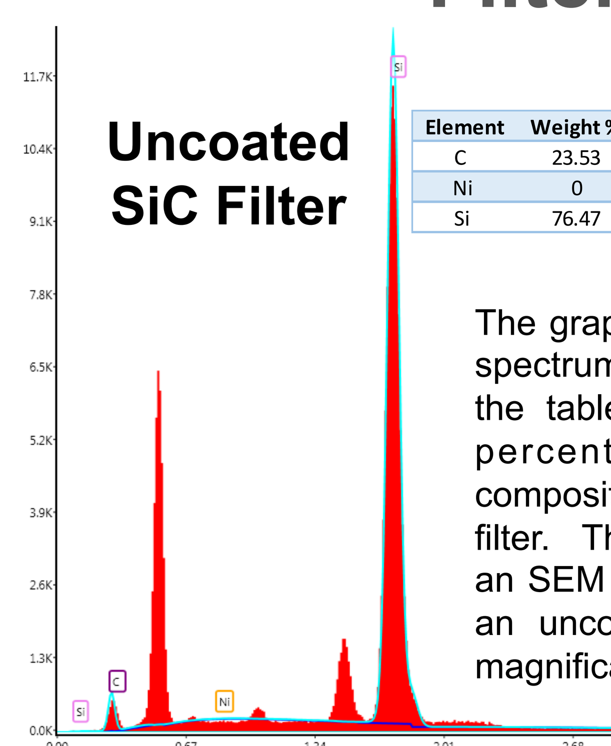


The image on the left shows a SiC ceramic foam filter before Electroless Deposition and the image on the right shows that same filter after the Nickel was Electroless Deposited.

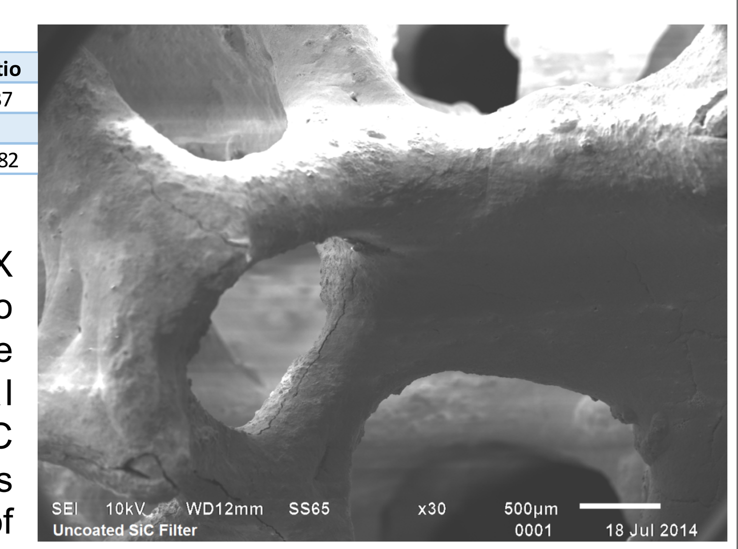


Filter Characterization

Uncoated SiC Filter

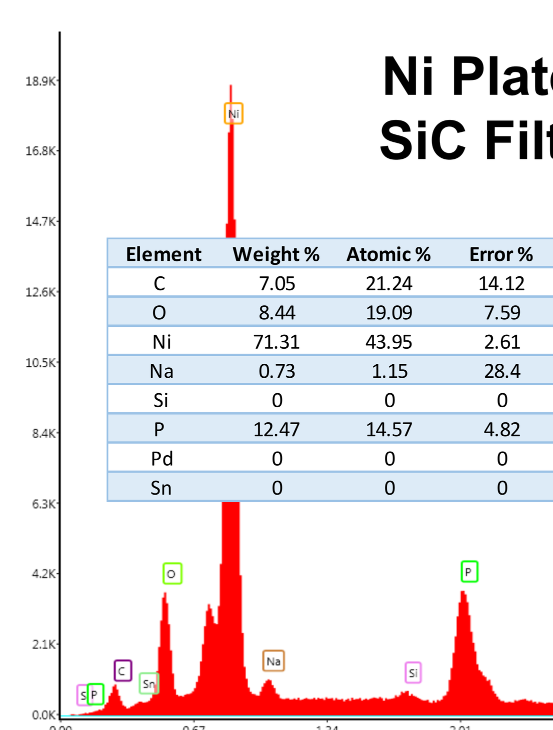


Element	Weight %	Atomic %	Error %	Net Int.	K Ratio
C	23.53	41.85	12.73	102.75	0.037
Ni	0	0	0	0	0
Si	76.47	58.15	2.46	3139.27	0.7282

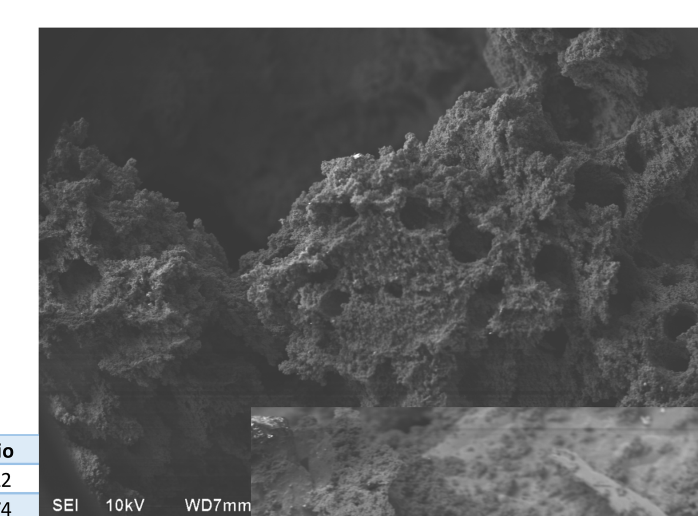


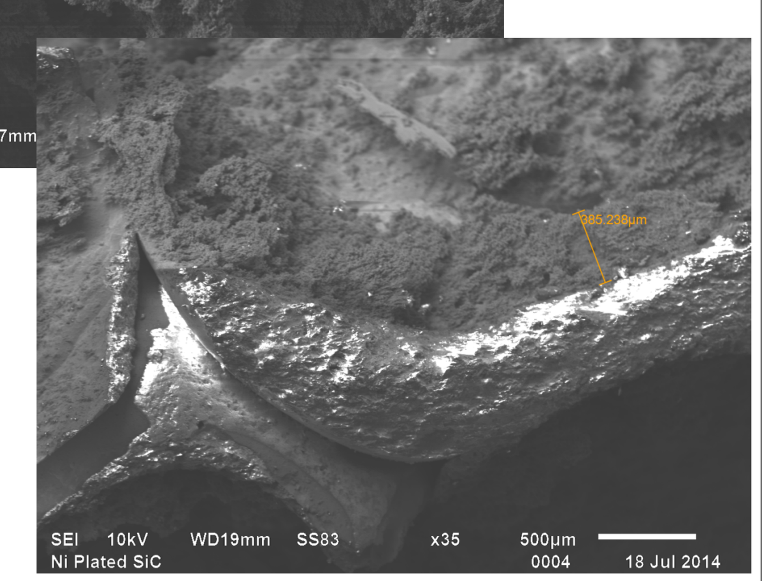
The graph on the left is an EDX spectrum that corresponds to the table above displaying the percentages and chemical composition of an uncoated SiC filter. The image on the right is an SEM image of the surface of an uncoated SiC filter at x30 magnification.

Ni Plated SiC Filter



Element	Weight %	Atomic %	Error %	Net Int.	K Ratio
C	7.05	21.24	14.12	45.27	0.0222
O	8.44	19.09	7.59	180.88	0.0574
Ni	71.31	43.95	2.61	992.38	0.6386
Na	0.73	1.15	28.4	9.98	0.0031
Si	0	0	0	0	0
P	12.47	14.57	4.82	267.82	0.1095
Pd	0	0	0	0	0
Sn	0	0	0	0	0





The graph on the left is an EDX spectrum that corresponds to the table above displaying the percentages and chemical composition of a Ni plated SiC filter. The two above images are SEM images of the surface (upper image) and cross-section (lower image) of a Ni plated SiC filter at x35 magnification.

Catalytic Regeneration Experimental Setup

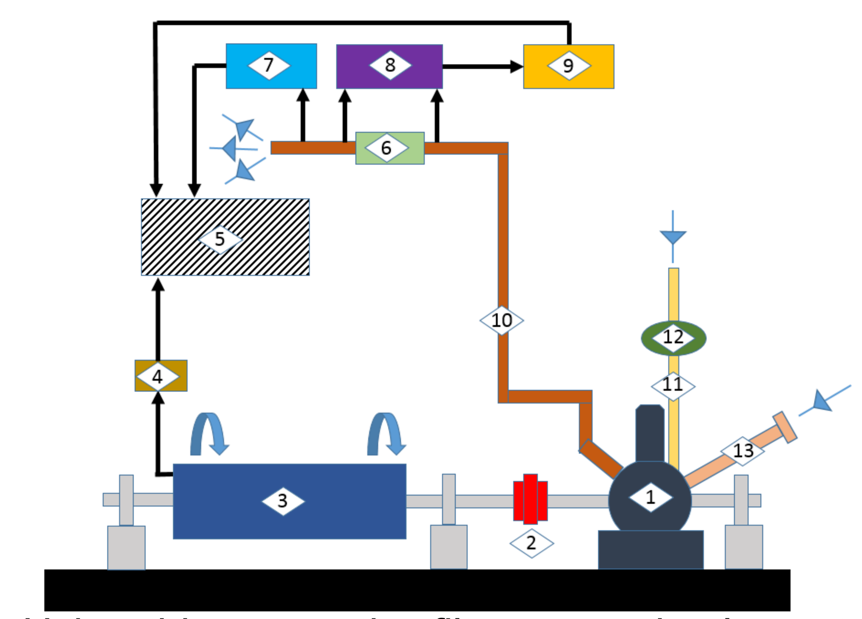
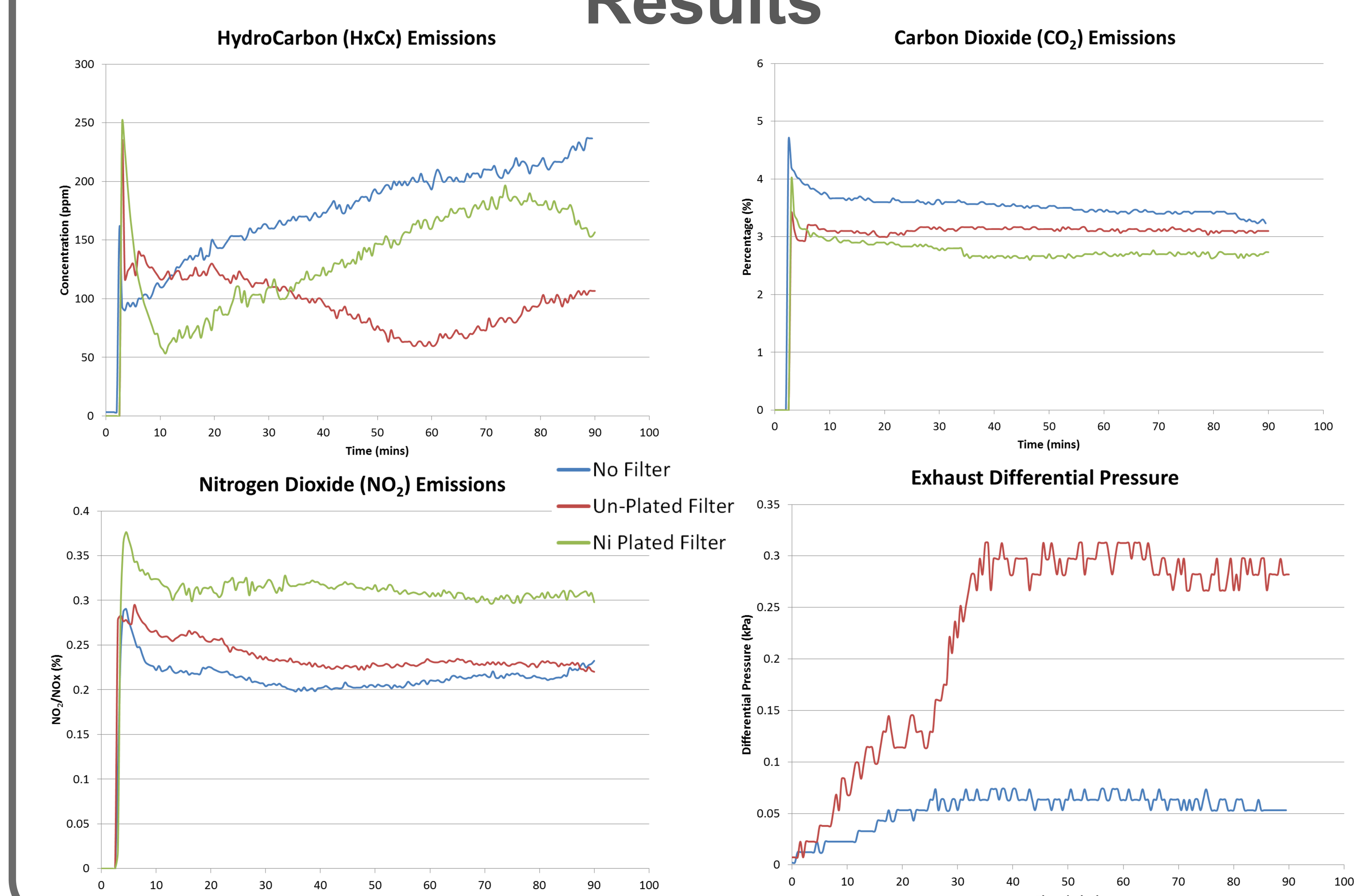


Figure Left showing: 1. Lister-Petter ADI (7.1 hp, 4-stroke, single-cylinder) diesel engine, **2.** Clutch, **3.** Dyno rotary drum, **4.** Dyno control unit, **5.** Computer, **6.** Filter, **7.** E instruments gas analyzer with CO_2, CO, O_2, HC, and NOx sensors, **8.** Kavlico P604 Differential Pressure Sensor, **9.** Data Logger, **10.** Exhaust pipe, **11.** Fuel Inlet, **12.** Flow-meter, **13.** Air inlet

Using this setup, the filters were implemented into our exhaust pipe and the engine was run using pure diesel fuel for 90 minutes with an inactivated clutch (idling) while collecting the CO_2, HC, NO_2, and Differential Pressure data.

Results



Conclusions

The SEM/EDX data confirms that Ni was Electroless Deposited onto the SiC filters surface at 3.2um/min. The chemical data confirms that SiC filters are sufficient in reducing the HCs and carbon soot since the HC values were lower and the differential pressure was higher than when no filter was present. The Ni Plated Filter NO_2 values were higher than the Un-plated, however the same trend could not be seen for CO_2. Therefore, we cannot confirm if Ni Plated SiC Filters efficiently catalyze the oxidation of carbon soot since the Differential Pressure Sensor became impaired during testing and we did not see the combined CO2 and NO2 concentration spikes in the exhaust.

Future Work

In the future different metal catalysts such as Cobalt, Iron, and Copper would like to be tested as well as testing these filters with different percentages of Biodiesel Fuel.

Acknowledgements

This work was funded by the Louisiana Board of Regents, through LASIGMA [Award Nos. EPS-1003897, NSF (2010-15)-RII-SUBR, and HRD-1002541].