CAAP Quarterly Report

Date of Report: *March 31, 2020*

Contract Number: 693JK31850012CAAP

Prepared for: *USDOT Pipeline and Hazardous Materials Safety Administration (PHMSA)*

Project Title: *Magnet-assisted Fiber Optic Sensing for Internal and External Corrosion-induced Mass losses of Metal Pipelines under Operation Conditions*

Prepared by: *Missouri University of Science and Technology (Missouri S&T)*

Contact Information: *Genda Chen, Ph.D., P.E., F.ASCE, Email: gchen@mst.edu, Phone: (573) 341-4462*

For quarterly period ending: *March 31, 2020*

Business and Activity Section

(a) General Commitments – Dr. Genda Chen directed the entire project and coordinated various project activities.

Dr. Chuanrui Guo, postdoctoral research fellow, and Mr. Ying Zhuo, Ph.D. in civil engineering at Missouri S&T, were on board for this project. They are responsible for the fabrication and characterization test of sensors under Dr. Chen's supervision.

(b) Status Update of Past Quarter Activities – Detailed updates are provided below by task.

Task 1 Optimization of a magnet-assisted hybrid FBG/EFPI sensor enclosed in a plexiglass container for simultaneous measurement of temperature and pipe wall thickness

Task 1 was completed by the fourth quarter of 2019.

Task 2 Development and validation of a graphene-based LPFG sensor with Fe-C coating for improved sensitivity in mass loss measurement in varying temperature environment

To investigate the evanescent field of a Graphene (Gr) coated LPFG, monolayer Gr was synthesized on a copper foil through a low pressure chemical vapor deposition (LPCVD) system as illustrated in Fig. 1(a). The system was assembled as shown in Fig. 1(b) in SPAR Lab, Missouri S&T. It consisted of three gas cylinders (CH_4, H_2 and A_r), a mass flow controller (MFC), a quartz tube (high temperature resistant), a high temperature furnace, a pressure control valve, a vacuum gauge and a vacuum pump.

The synthesis process started with flowing the H_2 and Ar into the quartz tube to anneal the copper (Cu) foil at 1030 °C for 30 minutes. This step was to remove the oxides on the copper surface. Then the CH_4 was flown into the chamber at 5 sccm (standard cubic centimeter per minute) for graphene growth. At this step, the CH_4 precursor was decomposed into carbon and hydrogen at 1030 °C and thus the carbon atom was deposited on the copper surface in the hexagonal form. As shown in Fig. 1(c), small flakes of

graphene emerged first and were then expanded into an integrated monolayer graphene on the copper surface. After 5 minutes of reaction, the chamber was cooled down to room temperature with the protection of H_2 and Ar.

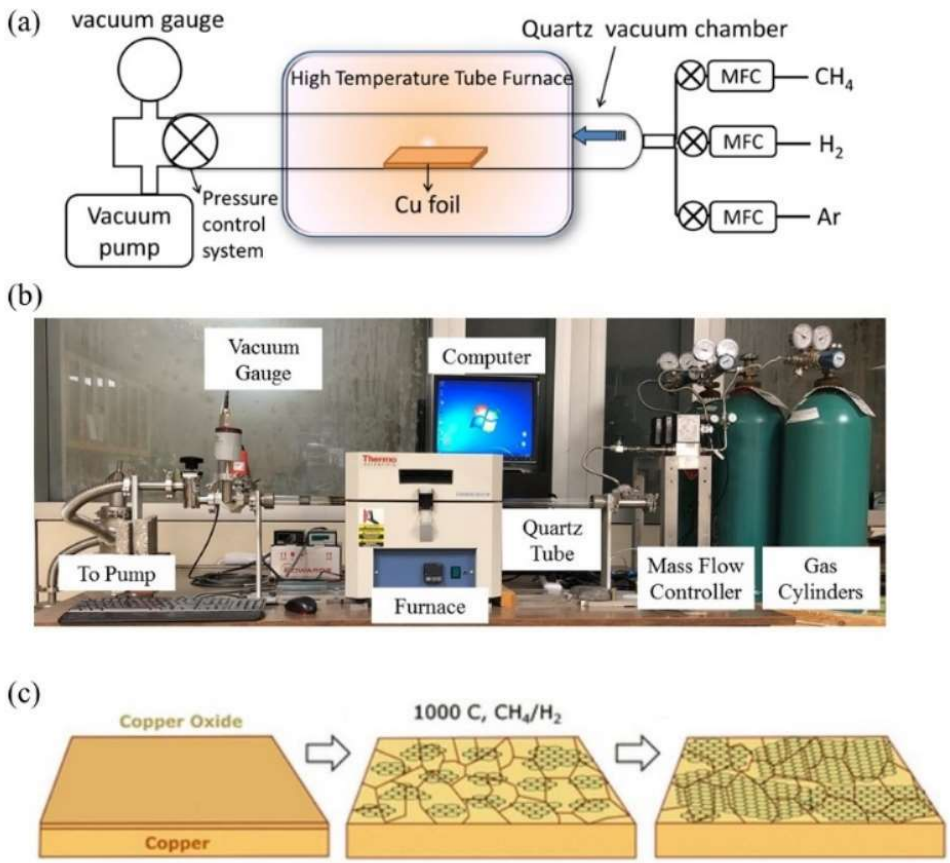

Fig.1 (a) Schematic illustration, (b) assembled LPCVD system, and (c) Graphene growth process on a copper foil

To characterize and validate the as-grown graphene, Raman spectroscopy was conducted on the Gr/Cu surface. The detected range of the Raman shift is from 1,200 to 3,000 cm^{-1}, which represents the typical resonant range of the carbon allotropy. Fig. 2 shows the measured Raman spectrum of the Gr/Cu sample. The G and 2D peaks indicate the existence of monolayer graphene on the copper foil.

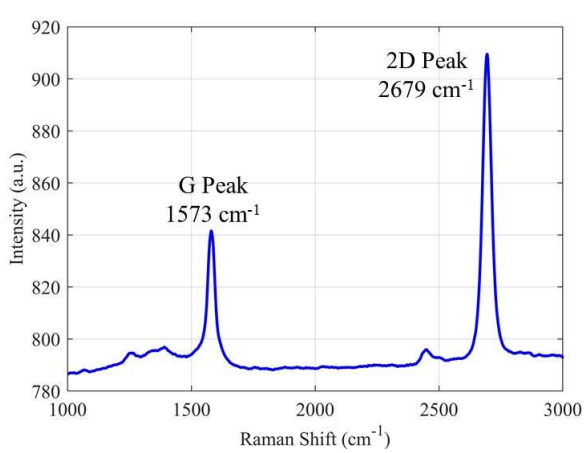

Fig. 2 Raman spectrum of as-grown graphene

After the synthesis of monolayer graphene on the copper foil, the graphene layer was transferred onto the LPFG surface for evanescent field study. Fig.3 illustrated the wet transfer process. A thin layer of Poly(methyl methacrylate) (PMMA, M_w=99,6000, Sigma Aldrich) was coated on the Gr/Cu sample as a "tape". This step was achieved by dropping the PMMA/chlorobenzene (46 mg/mL) solution on the Gr/Cu surface and spin-coating at 4000 rpm for 30 seconds. The coated sample was then heated at 140 °C for 10 minutes to evaporate the chlorobenzene and generate a uniform PMMA layer. To dissolve the copper foil, the sample was floated on the copper etchant solution for 4 hours to obtain the PMMA/Gr transparent film. The film was then cleansed by the deionized water twice to remove any possible ion residual. The LPFG fixed on a microscope slide was immersed under the film and lifted gradually to make sure the film fully covered the LPFG sensor. The residual water between the film and the LPFG was absorbed by a paper tissue at two ends, which created a pressurized adhesion by the atmosphere pressure. The sample was then heated at 60 °C to fully evaporate water and then increased to 140 °C to further enhance the adhesion between the Gr and the LPFG. An acetone bath was then conducted to remove the PMMA layer and thus obtain the Gr coated LPFG. Fig. 4 shows the sample at different steps.

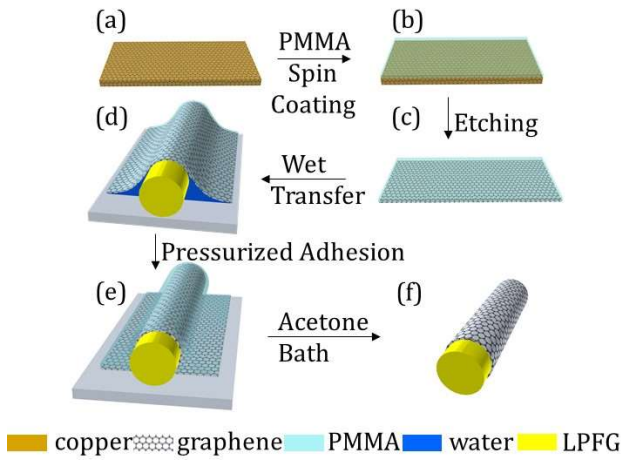

Fig.3 Graphene transfer procedure from PMMA to the LPFG surface

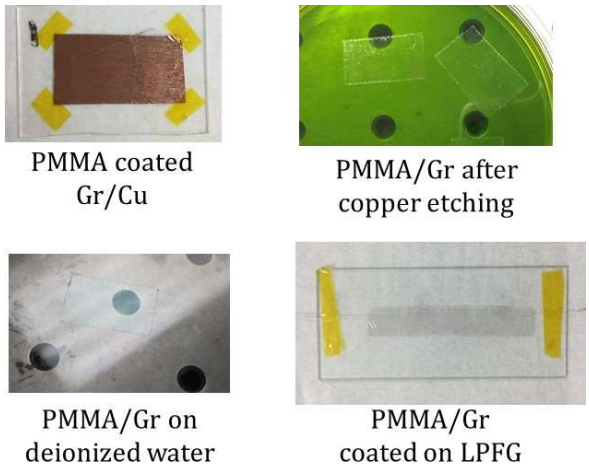

Fig. 4 Images of different steps of the transfer process

The above steps were repeated multiple times to stack multi-layer graphene on the LPFG surface. Transmission spectra of each case were acquired from the optical interrogator (Micron Optics Si255) as shown in Fig. 5.

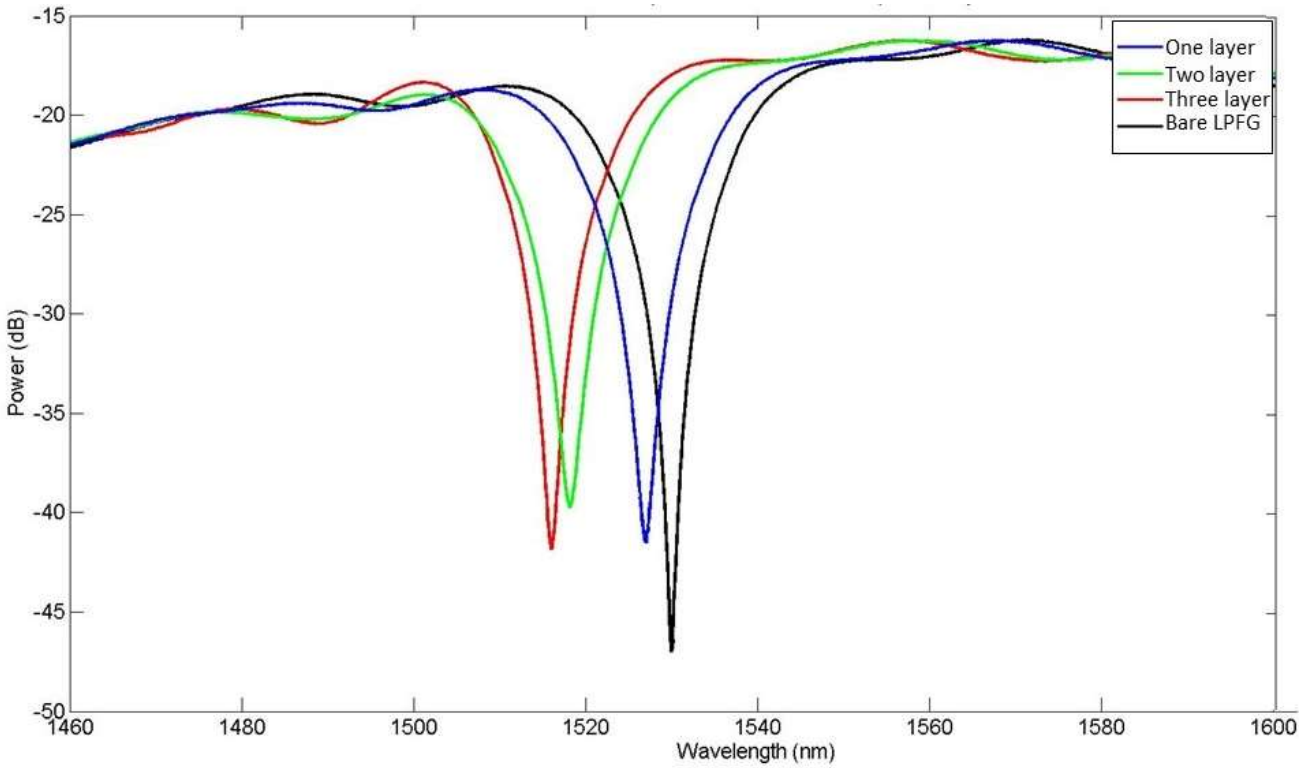

Fig.5 Transmission spectra of LPFG sensors coated with a different number of graphene layers

Task 3 Integration and field validation of multiple FBG/EFPI and multiplexed LPFG sensors for internal and external corrosion monitoring of a pipeline with temperature compensation.

This task will start from the 2st quarter in 2020.

(c) Planned Activities for the Next Quarter - The following activities in Task 2 and Task 3 will be executed during the next reporting quarter.

Task 2 Development and validation of a graphene-based LPFG sensor with Fe-C coating for improved sensitivity in mass loss measurement in varying temperature environment

The evanescent field attenuation will continue to be characterized once laboratory access is made available to the students after the coronavirus has been clear.

Task 3 Integration and field validation of multiple FBG/EFPI and multiplexed LPFG sensors for internal and external corrosion monitoring of a pipeline with temperature compensation.

The sensor package will be optimized for field applications and validated under various temperature levels.

(d) Problems Encountered during this Quarter and Potential Impact on Next Quarter – This project has been impacted by the COVID-19. At Missouri S&T, laboratories were closed to students on March 16, 2020. Since then, all experimental works stopped. Even computer access may become an issue due

to high traffic on internet during the period of virus spread, which will impact any computationally intensive tasks that require special computers from a remote site. Both the laboratory and computer access issues will likely continue to the first part or most part of the summer semester, which will impact the progress during the next reporting period.