Initiation of Chloride-Induced Corrosion of Low Carbon Steel Rebar in Concrete Using *In-situ* Quantitative Phase Microscopy

Ebenezer O. Fanijo and Alexander S. Brand

Civil and Environmental Engineering, Virginial Tech, Blacksburg, VA, USA

INTRODUCTION

Recent studies by Brand and colleagues [1-4] have used quantitative phase microscopy (QPM) to study *in situ*, nanoscale dissolution kinetics at mineral surfaces, as shown in Fig. 1. However, the application of QPM techniques is very limited in electrochemical studies. Thus, this study used this approach to quantify real-time nanoscale surface topography evolution during corrosion of steel surface using the unprecedented scope of spectral modulation interferometry (SMI). From this experiment, high quality quantitative kinetic data and analysis can be obtained on the fundamental micro- and nano-structural mechanisms and initiation of steel corrosion.
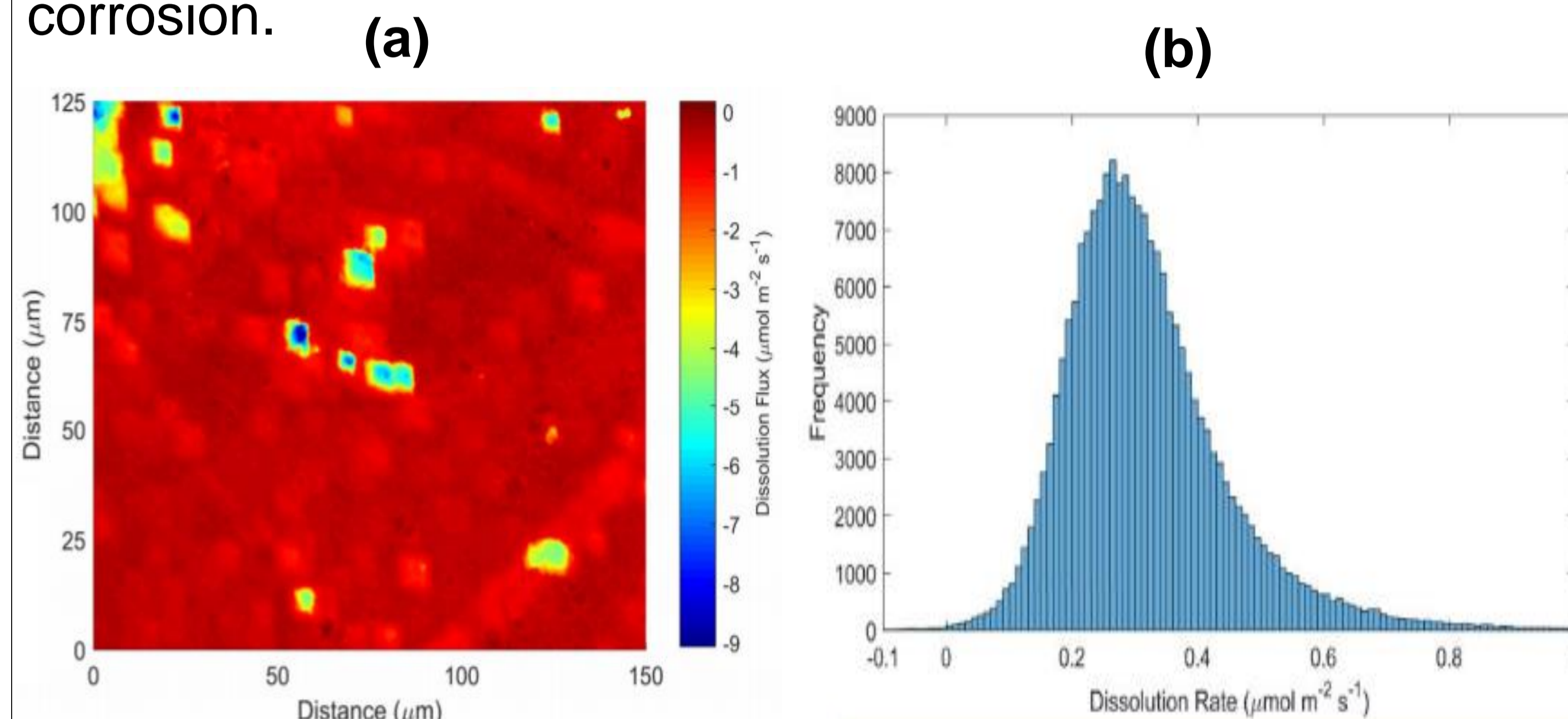

Fig. 1. (a) spatial and (b) probabilistic distribution of mineral surface dissolution rates [4].

OVERVIEW

Spectral modulation interferometry (SMI) is a novel QPM technique developed by Zhu's team at Virginia Tech [5]. The SMI offers sub-nanometer vertical resolution and high-speed, speckle-free phase imaging using a Linnik interferometer (Fig. 2). After reflecting off the sample surface, the object beam is recombined with the reference beam and the interference spectrum is recorded by a custom spectrometer and a linescan camera. In this configuration, a single spectrum contains of spatial, temporal, or spectroscopic information. The rapid collection of temporally and spatially resolved phase information provides the necessary information to reconstruct dynamic 3D surface topography evolution.
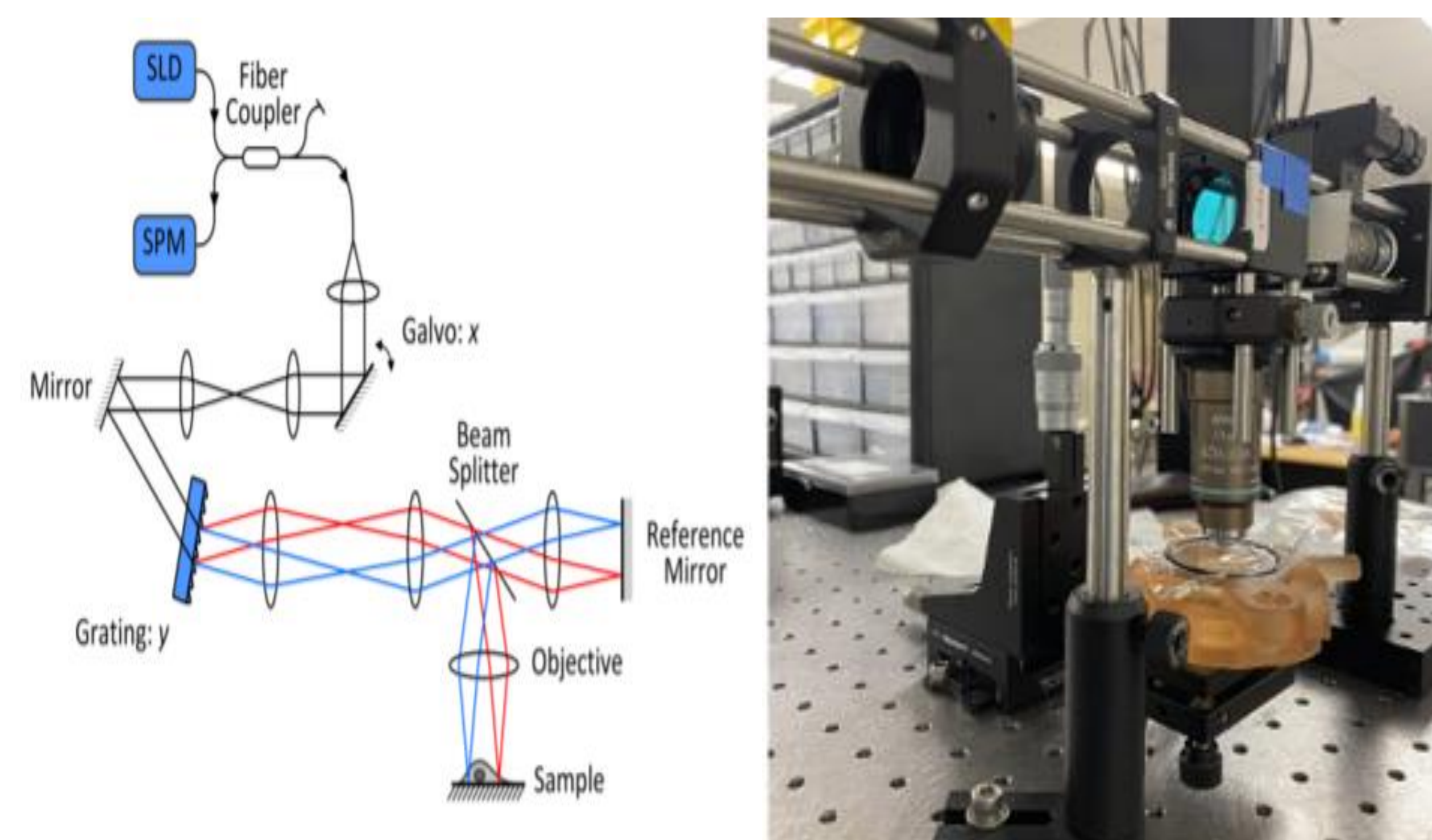

Fig. 2. Schematic of the SMI technique

MATERIAL AND METHOD

ASTM A615 steel rebar (a low carbon steel) was used in this study. Before the corrosion test, steel surfaces was polished to avoid the effect of surface roughness on corrosion. To study the *in situ*, real-time nanoscale dissolution kinetics of corrosion under static and flowing conditions, a preliminary fluid reaction cell was designed and constructed, which is a novel outcome from this research study. The fluid cell is specially designed to incorporate the working electrode (steel sample), silver-silver chloride (Ag/AgCl) reference electrode and platinum wire reference electrodes as well as space for the SMI objective lens (Fig. 3). With this fluid cell, both *In situ* nanoscale corrosion (SMI) and electrochemical (Gamry 1010E potentiostat) monitoring are performed simultaneously.
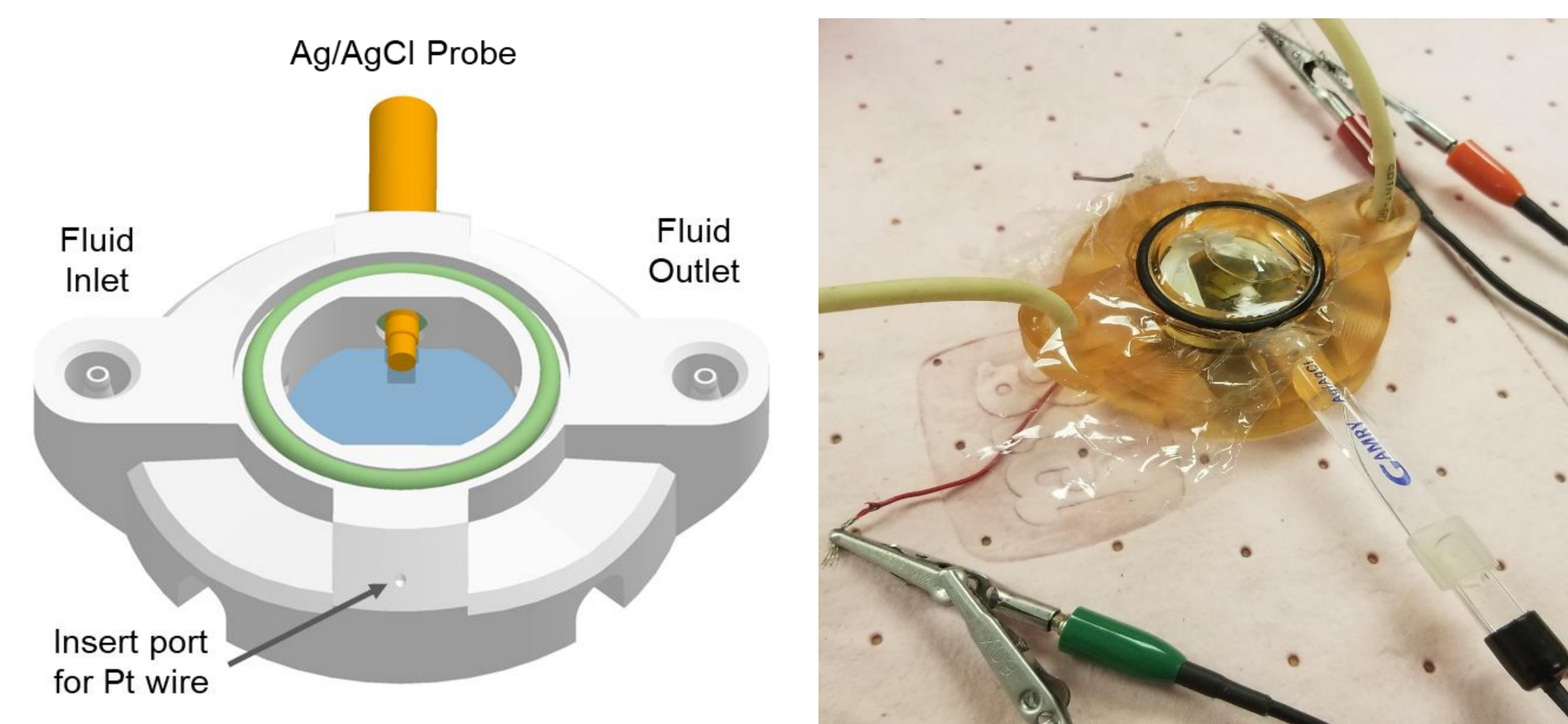

Fig. 3. Prototyped of the developed fluid cell

With the SMI test, electrochemical studies were first conducted to evaluate the effects of static and flowing solution (0.5 wt% NaCl) on the linear polarization resistance (LPR) and potentiodynamic polarization (PDP) test following ASTM G102. Prior to LPR and PDP experiments, the specimens were exposed to the solution, and held at the open circuit potential (OCP) for one hour.

PRELIMINARY RESULTS

The OCP of steel sample at static and flowing condition of 40ml/min in a 0.5 wt% NaCl solution is shown in Fig. 4. Generally, the curves at both static and 40mL/min flow rate have almost similar features with a stable state OCP over time. Also, LPR and PDP results for the steel sample at static and 40mL/min flow rate are shown in Fig. 5a-d. Three LPR and PDP curves were evaluated and plotted to show the performance of the fluid cell.
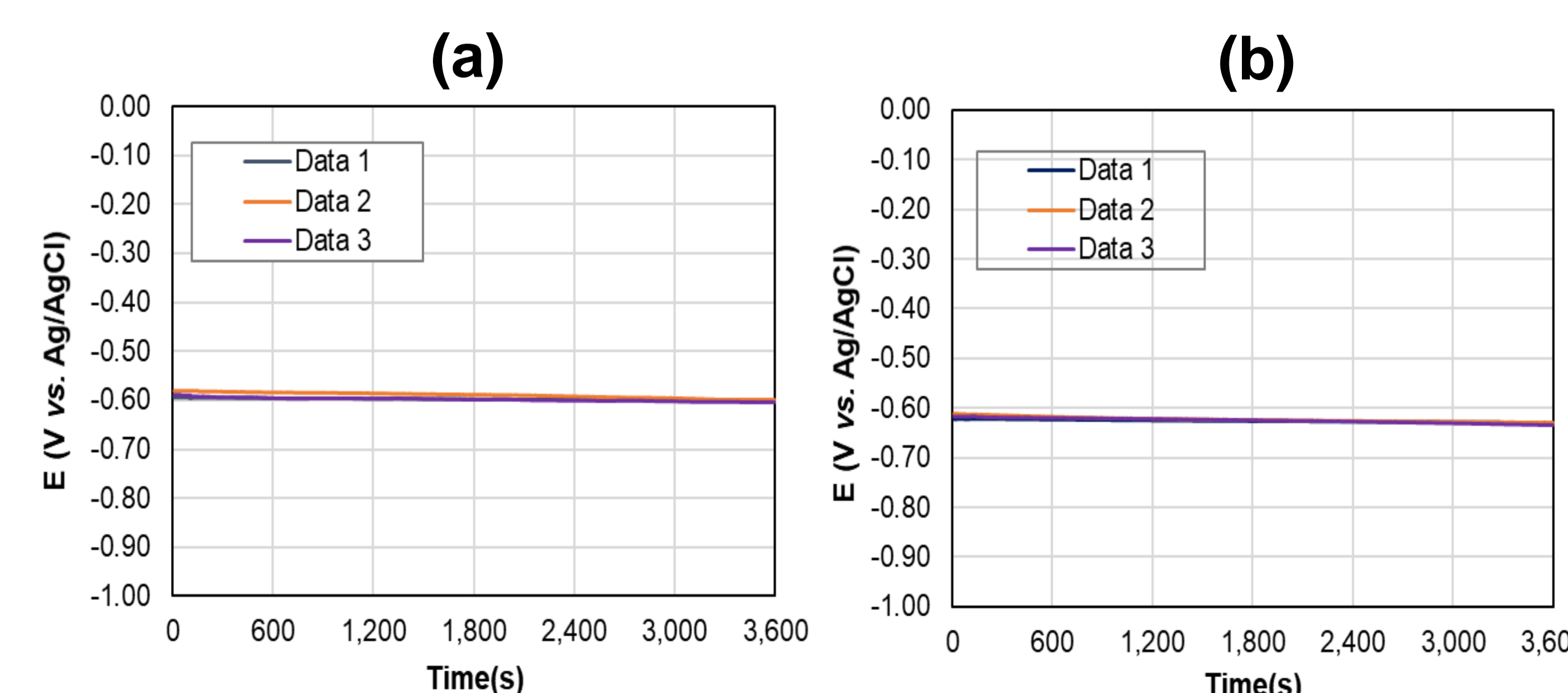

Fig. 4. OCP at (a) static condition, (b) 40mL/min flowing condition

PRELIMINARY RESULTS CONT'D

It can be deduced that the fluid cell configuration yields repeatable results for the OCP and electrochemical (LPR and PDP) experiments.
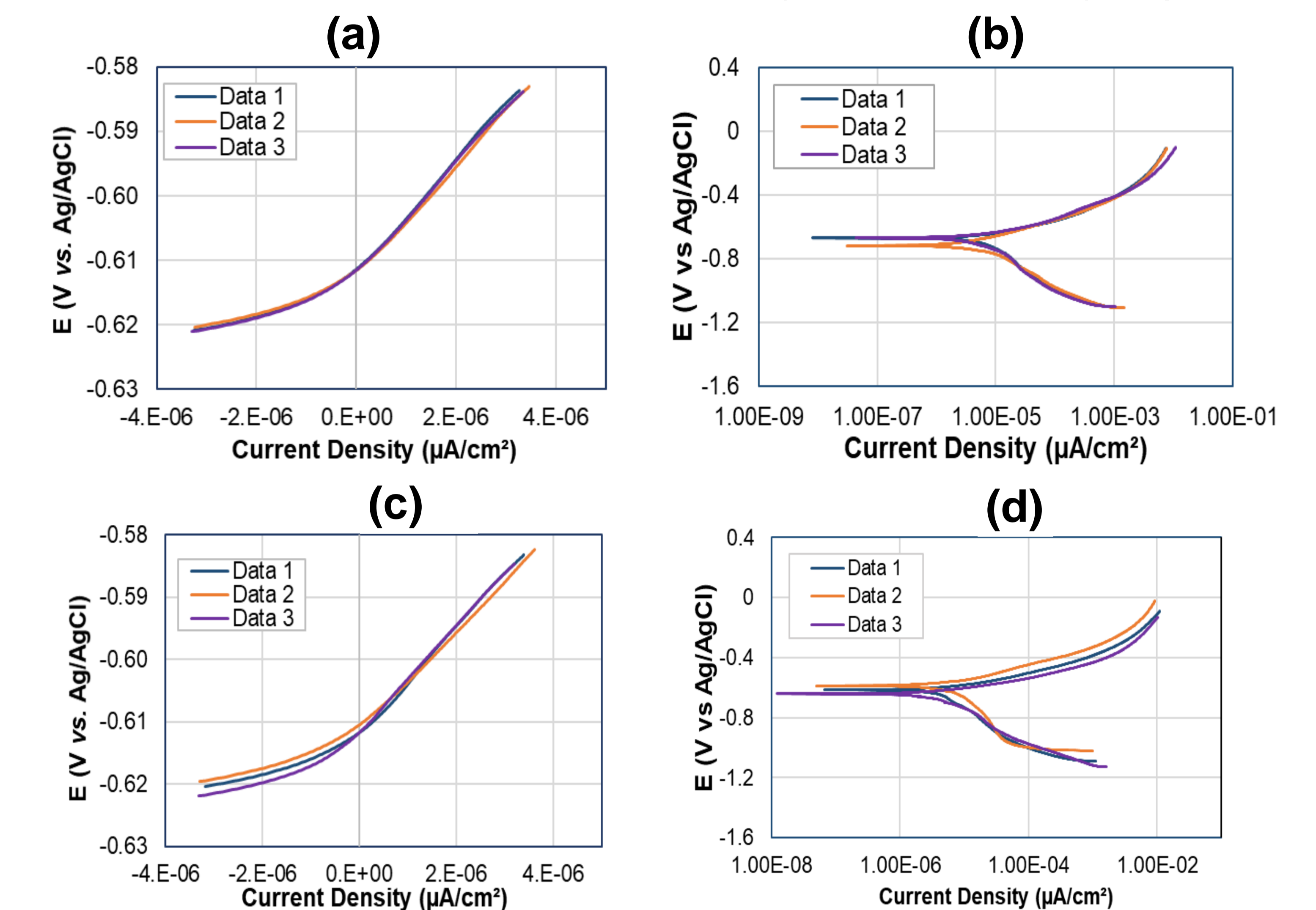

Fig. 5. Electrochemical test for steel sample (a) LPR at static, (b) PDP at Static, (c) LPR at 40mL/min, (d) PDP at 40mL/min flow rate

SMI imaging were acquired before and after electrochemical corrosion test. Fig. 6 showed the 3D topography images of the uncorroded and corroded steel surface with the grain boundaries clearly distinguishable. It can deduced that SMI technique can predict corrosion behavior of sample surface over time, with grain boundaries increasing from ±30nm (for the uncorroded sample) to ±300nm.
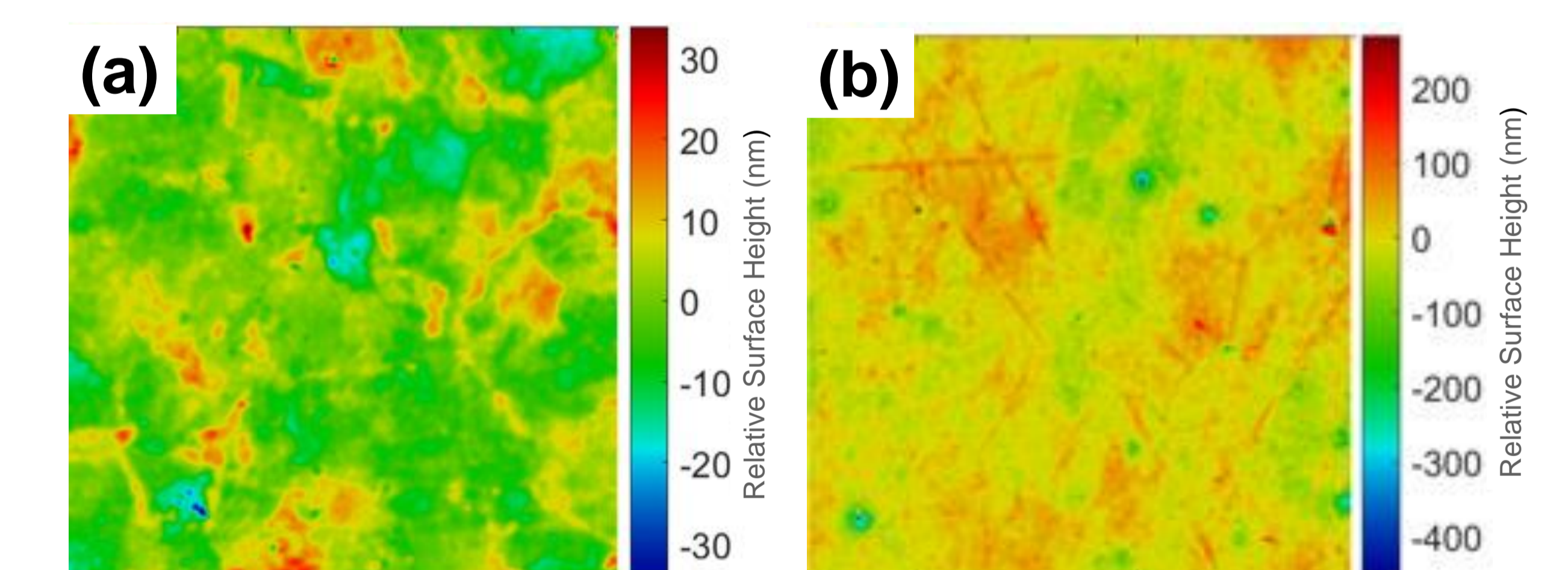

Fig. 6. Typical SMI images (a) before (b) after corrosion experiment

CONCLUSION AND FUTURE WORK

The preliminary LPR and PDP result (Fig. 5) showed the performance of the novel fluid cell developed for this study that could yield repeatable results. With the fluid cell, the surface topography images (Fig. 6) acquired before and after corrosion using the unprecedented SMI deduced that the techniques can predict the corrosion mechanism of low carbon steel sample. Future experiments will use the SMI to characterize the mechanisms and kinetics related to initiation and propagation of chloride-induced corrosion.

REFERENCES & ACKNOWLEDGEMENTS

1. Brand, A.S., et al. *Cem Concrete Res*, 130, 2020, 105989.
2. Brand, A.S., et al. *Cem Concrete Res*, 118, 2019, 69–83.
3. Brand, A.S., and J.W. Bullard. *Langmuir*, 33(38), 2017, 9645-9656.
4. Brand, A.S., et al. *Geochim Cosmochim Acta*, 213, 2017, 317-329.
5. Shang, R., Chen, S., Li, C., & Zhu, Y. *Biomedical optics express*, 6(2), 2015, 473-479.