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# A literature review of in situ transmission electron microscopy technique in corrosion studies

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ABSTRACT

One of the biggest challenges in corrosion investigation is foreseeing precisely how and where materials will degenerate in a designated condition owing to scarceness of accurate corrosion mechanisms. Recent fast development of in situ transmission electron microscopy (TEM) technique makes it achievable to better understand the corrosion mechanism and physicochemical processes at the interfaces between samples and gases or electrolytes by dynamical capture the microstructural and chemical changes with high resolution within a realistic or near-realistic environment. However, a detailed and in-depth account summing up the development and latest achievements of in situ TEM techniques, especially the application of emerging liquid and electrochemical cells in the community of corrosion study in the last several years is lacking and is urgently needed for its heathy development. To fill this gap, this critical review summarizes firstly the key scientific issues in corrosion research, followed by introducing the configurations of several typical closed-type cells. Then, the achievements of in situ TEM using open-type or closed-type cells in corrosion study are presented in detail. The study directions in the future are commented finally in terms of spatial and temporal resolution, electron radiation, and linkage between microstructure and electrochemical performance in corrosion community.

## 1. Introduction

Corrosion is defined as not only the destructive oxidation of metallic materials but also the degradation of nonmetallic materials and their attendant loss of function owing to a chemical or electrochemical interaction with environment (Li et al., 2015; National Research Council, 2011). Corrosion is common and results in innumerable disasters and hundreds of billions of dollars loss annually, such as the explosion of oil pipeline in the Chinese cities of Qingdao and Kaohsiung in November 2013 and August 2014, respectively, which caused totally 94 deaths and 457 injuries (Jungjohann et al., 2016; Li et al., 2015; Scully and Harris, 2012). A recent survey by Hou et al. points out that the total loss of corrosion, estimated by Uhlig's method, is approximately US$310 billion in China in 2014 (Hou et al., 2017). Globally, the cost of corrosion is over US$4 trillion annually, which is about 40 times damages in Hurricane Katrina (Li et al., 2015).

Practical application in electrical energy conversion and storage technologies, and nanotechnology is also restricted because of corrosion. For example, the local anodic corrosion of aluminum foil in cathode current collector not only shorten the lifetime of current 4 V lithium ion batteries (LIBs) but also remains a roadblock limiting the development of next-generation 5 V LIBs (Wang et al., 2017a). The dispersed platinum- or platinum-alloy-based electro-catalysts on a high-surface-area carbon support in proton exchange membrane fuel cells (PEMFC) undergo oxidation and dissolution obviously due to the dimensions of the catalysts reaches to nanometer scale, leading to degradation of the PEMFC performance with time (Cherevko et al., 2016; Jiang and Chu, 2014; Shan et al., 2018). Cadmium sulfide (CdS) powders and electrodes are one of the most promising photocatalyst for hydrogen generation and organic dye decomposition owing to its excellent light absorbing characteristics (DiMeglio and Bartlett, 2017). Corrosion of CdS in an aerated aqueous solution usually causes to formation of water-soluble cadmium sulfate, surface dissolution, and failure of catalytic activity (DiMeglio and Bartlett, 2017).

However, unlike weather-related disasters (e.g., earthquakes, tornadoes, and tropical storms), corrosion can be controlled though long-term prediction is extreme challenge and it cannot be eliminated completely (Gin et al., 2017; Li et al., 2015). Half of the corrosion costs may be prevented and controlled assuming appropriate corrosion management and methods, such as a maintenance approach which is

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called "anticipate and manage", are applied (Li et al., 2015). The most usually adopted strategies for preventing and controlling corrosion include but not limited to organic and metallic coatings, corrosion resistance alloys, polymers, corrosion inhibitors, cathodic protection, glass and ceramics, and control of the environment, etc. (Gan et al., 2017; Li et al., 2017a; National Research Council, 2011; Xie et al., 2015; Zhang et al., 2018). For improving the corrosion protection efficiency of the abovementioned methods, it needs comprehensive understanding of all related factors (e.g., compositions and microstructures of the substance, the external conditions including relative humidity, pH, temperature, stress, and salinity, etc.) which may result in degradation (Li et al., 2015). The synergism among these relevant factors further augment the complexity of degradation processes. Accurate prediction of corrosion degradation of materials in actual service environments becomes one of the largest challenges in corrosion investigation (Li et al., 2015).

In situ transmission electron microscopy (TEM) techniques supply knowledges not obtainable by other approaches (Liao and Zheng, 2016; Wu et al., 2016). It enables imaging of corrosion/dissolution processes of metal and alloys over time under realistic or near-realistic reaction conditions (Bettini et al., 2012; Jiang et al., 2017; Yang et al., 1997). Achievements in the area of in situ TEM in recent several years have brought material characterization methods to the position where these fundamental problems may be interpreted further sufficiently (Janish et al., 2016). More recently, in situ electrochemical TEM has been further proposed as a strong instrument to study the dynamic chemical or electrochemical reactions, especially at the early stage of materials degradation at atomic and nanometer scales in real-time under given conditions (Cuong et al., 2018; Liu et al., 2018a). A search of the Web of Science Core Collection for "in situ TEM" plus its full name "in situ transmission electron microscope (microscopy)" in topic with years range from 1998 to 2017 yields more than 2600 scientific papers. The distribution over the last 20 years is depicted in Fig. 1. As shown in Fig. 1, the annual publication increases over time, and reaches to 290 in the year 2017. The significant increase in the last 6 years indicates the importance and speedy growing of in situ TEM, and the study of in situ TEM technique itself and its application in different areas have become a hot topic. However, previous reviews regarding in situ TEM techniques are mainly focused on the optimization of in situ TEM holder and the progresses of in situ TEM in the areas of energy conversion and storage, especially in the studies of LIBs (de Jonge and Ross, 2011; Harks et al., 2015; Liu and Huang, 2011; Liu et al., 2012; Wu and Yao, 2015; Xie et al., 2017b). Review literature for the applications and achievements of in situ TEM techniques in the investigation of corrosion is scare to date.

In this review, we will first summarize the key scientific problems in the corrosion research. Then, configurations of several typical closed-type in situ TEM cells will be introduced, followed by achievements of in situ TEM techniques in corrosion in recent years. Considering that

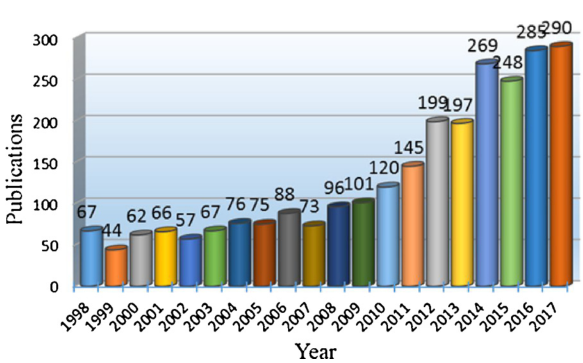


**Fig. 1.** An ever-growing number of scientific publications involving investigation/application of in situ TEM in the past 20 years based on the search results from Web of Science Core Collection (Until December 2017).

dry corrosion, stress crack corrosion, dislocation, etc. also belong to corrosion or play a critical role in corrosion initiation, to interpret the fundamental corrosion mechanism of a metal or metal alloy, the achievements of in situ TEM observation of metallic oxidation, phase-transformation, dislocation, precipitate characteristics, etc. under conditions without water or moisture are also partly included. Finally, challenges and perspectives using in situ TEM techniques to study material degradation will be given for design more powerful cell, better understanding the mechanism of corrosion especially in the early stage, and thereby develop tactics to decrease corrosion-related losses in the future.

## 2. Challenging scientific issues in corrosion research

There are many forms of corrosion and they can be generally divided into uniform corrosion and localized corrosion in terms of corrosion morphologies (Chee and Burke, 2016). In most cases, localized corrosion plays a more dominant role. Localized corrosion commonly occurs in the form of stress corrosion cracking (SCC), pitting, galvanic corrosion, crevice corrosion, as well as intergranular and exfoliation corrosion (Bertali et al., 2016, 2017; Burke et al., 2018; Frankel and Sridhar, 2008; Harlow and Taheri, 2015; Jiang and Devine, 2016; National Research Council, 2011). Localized corrosion of metals and alloys because of breakdown of passivity by aggressive species, such as $Cl^{-}$, is an elementary corrosion process but is inadequately interpreted (Frankel et al., 2017; Frankel and Sridhar, 2008). For example, it has been assumed that selective attack is owing to defects in the oxide film, but the reasons of passive layer failure and pit formation in initial stages remain elusive (Chee and Burke, 2016; Harlow and Taheri, 2015). The most important aspects to control localized corrosion susceptibility have been argued extensively over the last decades and there also remains disagreement among the pioneers in the area of corrosion (Frankel et al., 2017).

Many traditional electrochemical measurements (such as potentiodynamic polarization curves (Li et al., 2018; Liu et al., 2018b; Wang et al., 2017b; Xie and Shan, 2018; Xie et al., 2015), cyclic polarization (Chee and Burke, 2016), electrochemical impedance spectroscopy (EIS) (Bland et al., 2017a, b; Li et al., 2018; Xie et al., 2017a), and electrochemical noise (Bolivar et al., 2017)), and ex-situ modern characterization techniques (such as TEM (Jiang and Devine, 2016; Wang et al., 2017b; Zhang et al., 2015), scanning electron microscope (SEM) (Patrick et al., 2015; Zhang et al., 2017a), X-ray photoelectron spectroscopy (XPS) (Gan et al., 2017)) are employed to estimate the corrosion resistance and analyze the micromorphology/microstructure of a metal and alloy system. These conventional electrochemical methods are difficult to elucidate the reason of corrosion essentially, and the post-mortem characterization techniques cannot observe the changes of system in real-time during corrosion, though the conventional electrochemical methods can macroscopically reflect the capability of corrosion protection for a material. To continuous document these changes in morphology, structure, and chemical composition during degradation of a material, especially in the initial stage, many in situ techniques are proposed. In situ approaches pertaining to corrosion in recent years include electrochemical impedance spectroscopy (Couet et al., 2017; Grassini et al., 2018), scanning electrochemical microscopy (SECM) (Filotás et al., 2017), scanning Kelvin probe (SKP) (Thomson and Frankel, 2017), atomic force microscopy (AFM) (Birbilis et al., 2009), combined AFM-SECM (AFM-SECM) (Izquierdo et al., 2017; Zhang et al., 2014), electrochemical scanning tunneling microscopy (ECSTM) (Martinez-Lombardia et al., 2014), inductively coupled plasma-mass spectroscopy (ICP-MS) (Lutton et al., 2017), electrochemical surface forces apparatus (EC-SFA) (Merola et al., 2017), optical microscope and digital image correlation (DIC) (Bettini et al., 2012; Bolivar et al., 2017; Wang et al., 2016), environmental scanning electron microscopy (ESEM) (Mortazavi et al., 2015), infrared reflection absorption spectroscopy (IRRAS) (Persson et al., 2013), atomic emission

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