



Sensing of the dynamic concentration field at the solid/liquid interface using a Mach–Zehnder interferometer

Boyu Yuan^{a,b}, Wei Li^{a,*}, Chao Wang^{c,**}, Liang Li^c

^a School of Mechanical and Electrical Engineering, China University of Mining and Technology, Xuzhou, Jiangsu 221116, China

^b School of Physics and Electronic Engineering, Jiangsu Normal University, Xuzhou, Jiangsu 221116, China

^c School of Chemistry and Chemical Engineering, Jiangsu Normal University, Xuzhou, Jiangsu 221116, China

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ABSTRACT

A Mach–Zehnder interferometer is employed to study the dynamic concentration field within diffusion layer in transparent liquid solutions at the solid/liquid interface. The method is applied to investigate the electrodeposition of nickel in 0.20 mol dm^{-3} NiSO_4 solution. Interferograms of an object wave through an experimental cell containing diffusing solutions are recorded continually by a CMOS image sensor and displayed on a monitor in real time. The two-dimensional concentration change of the solution at the solid/liquid interface is determined according to the interference fringes. Software is developed to obtain the dynamic concentration field automatically. The calculated results match well with theory values. Its ease of fabrication offers the attractive applications in chemical and biological sensing.

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1. Introduction

Generally speaking, diffusion is the movement of a species under the influence of a concentration gradient in solution [1]. Diffusion and diffusion layer have important influence on wet-chemical reaction system. Study of concentration field with diffusion layer is a crucially important subject in the field of chemistry both from fundamental and technical standpoints. Knowledge of concentration field within diffusion layer is useful to understand reaction route, electro-dissolution, electro-deposition, diffusion and convection near electrode, and so on [2–5]. Moreover, the study of concentration field is also important in biology, materials, medicine, fluidics and other domains [6–13].

Many techniques have been explored to study the concentration profile within diffusion layer of solution, such as the refractometry [11], spectroscopy [14,15] and interferometry [4,16–18]. Optical methods provide a convenient and noninvasive tool for studying the concentration field and continue to shed light on dynamic processes. The interferometric technique is one of the most widely used techniques for concentration field study because it is a whole-field technique. Leger et al. used phase-shift Mach–Zehnder interferometer to first obtain quantitative proof of the diffusive character of the mass transport in thin gap electrodeposition

experiments [4]. Anand et al. determined the diffusion coefficients automatically by use of digital holographic interferometry [17]. The convenient method can be suitable for continuous monitoring the diffusion process with high accuracy. Recently, You et al. employed a Michelson interferometer to measure the concentration profile within diffusion layer in a galvanic displacement reaction system [18]. It was proved to be a useful tool to further confirm the validity of theory model deduced by other electrochemists. During most electrochemical reactions, the diffusion layer changes with the wave of the concentration near the solid/liquid interface. For fast, quantitative and simultaneous measurements of the dynamic concentration field and the diffusion layer thickness, more suitable methods should be explored to give new insights into those processes.

In this paper, a Mach–Zehnder interferometer is employed to investigate the two-dimensional concentration changes of transparent liquid solutions at the solid/liquid interface during electrochemical reactions. It can provide *in situ* measurement of full-field concentration changes at the interface during electrochemical processes without external effect on most electrochemical reactions. The resolution and measurement range of the interferometer can be adjusted in a wide range. With these advantages, it is well suited for a detailed study of mass transport processes in transparent media. Digital image/video processing methods have been used to analyze the interferogram video sequences obtained by the interferometer. The software for determination of the concentration distribution has been developed. The dynamic concentration changes in nickel electrodeposition

* Corresponding author. Tel.: +86 51683590798; fax: +86 51683590798.

** Corresponding author. Tel.: +86 51683403003; fax: +86 51683403003.

E-mail addresses: byww2004@163.com (W. Li), wangc@jsnu.edu.cn (C. Wang).

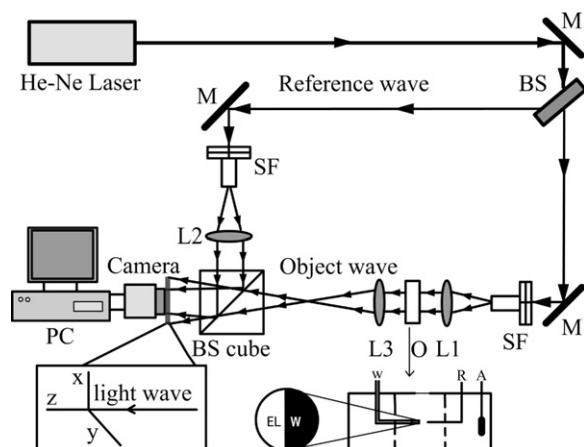


Fig. 1. The experimental set of the holographic recording system: M, mirror; BS, beam splitter; SF, spatial filter; L1 and L2, collimating lens; O, experimental cell; L3, object lens.

have been visually presented. The interferometer is proved to be useful and convenient to investigate the dynamic changes at the solid/liquid interface during electrochemical processes. The experimental setup and theoretical analysis of the method are introduced and discussed in detail.

2. Materials and methods

2.1. Optical setup

Fig. 1 shows the experimental set of the holographic recording system. It consisted of an improved Mach–Zehnder interferometer. Light from a He–Ne laser source was split into two beams using a beam splitter. Each beam was expanded and collimated with a microscopic objective and a collimating lens. One beam passing through the experimental cell was the object beam. The other acted as reference beam. They interfered at a CMOS image sensor forming the interferograms. A personal computer was connected to the image sensor for recording the interferograms at 25 frames per second.

2.2. Electrochemical system

The electrochemical cell is the same as that [19]. It contained a three-electrode system. An electrode of copper rod (2 mm diameter, 99.9%) was employed as the working electrode. The entire electrode was sealed with a thin layer of epoxy resin in a glass tube, with the end of the copper rod exposed to the solution. The counter electrode was a large sheet of nickel. A saturated calomel electrode (SCE) was employed as the reference electrode. A Luggin capillary was also employed between the working electrode and the reference electrode. Before each experiment, the Cu electrode was mechanically polished with emery paper and then cleaned by alcohol and triply distilled water in an ultrasonic bath. The electrolyte was 0.20 mol dm^{-3} NiSO_4 solution, which was prepared from reagents of analytical grade and triply distilled water. The galvanostatic electrodeposition of the nickel was performed by a CHI660B electrochemical workstation (CHI Instruments Inc.). The cathodic current density was kept at 1.91 mA cm^{-2} . All measurements were carried out at room temperature ($20 \pm 0.5^\circ \text{C}$). Fig. 2 shows the V – t curve of the galvanostatic electrodeposition of the nickel in 0.20 mol dm^{-3} NiSO_4 solution.

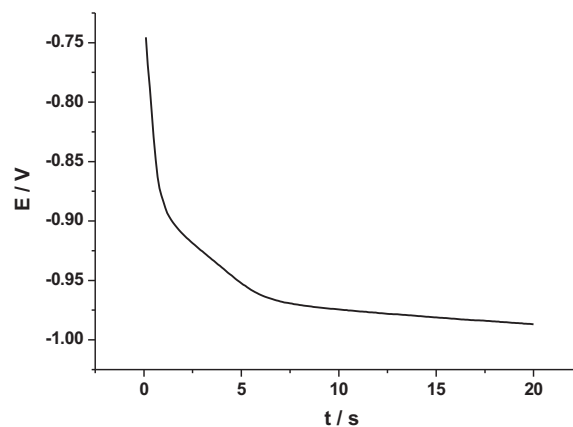


Fig. 2. The V – t curve of the galvanostatic electrodeposition of the nickel in 0.20 mol dm^{-3} NiSO_4 solution.

2.3. Image processing

2.3.1. Determination of the phase difference

For obtaining the phase maps conveniently, carrier fringes were introduced by moving the object lens L3 in Fig. 1. The carrier wave was obtained by a slight displacement of L3 along with x or y axis (depend on the location of interface). In the present experiment, the solid/liquid interface was vertical (along with y axis), thus the displacement was horizontal (along with x axis). Fig. 3 shows two interferograms and the corresponding Fourier spectra with and without the carrier fringes. Because the light was shaded by the copper electrode, it presented a dark shadow at a half part of the interferograms.

In experimental system of digital recording, the fringe pattern of the form can be found as,

$$g(x, y, t) = a(x, y) + b(x, y) \cos [2\pi f_0 x + \phi(x, y, t)] \quad (1)$$

where the time-varying phase $\phi(x, y, t)$ contains the desired information; $a(x, y)$ and $b(x, y)$ represent irradiance variations arising from the non-uniform light transmission by the experimental cell; f_0 is the spatial frequency of the fringe carriers given by the slight horizontal displacement of the object lens L3.

In the experiment, the concentration at the solid/liquid interface changed as the electrochemical reactions carried out, which would cause the change of the refractive index of the solution. The interference fringes in the interferograms deformed accordingly. The changes in the phase ($\phi(x, y, t)$) were brought about by the variations of the refractive index inside the electrochemical cell.

If the recording is made with identical conditions of illumination, the intensity $g(x, y, t)$ recorded on the interferograms at different times t_1 and t_2 are given by,

$$g(x, y, t_1) = a(x, y) + b(x, y) \cos [2\pi f_0 x + \phi(x, y, t_1)] \quad (2)$$

$$g(x, y, t_2) = a(x, y) + b(x, y) \cos [2\pi f_0 x + \phi(x, y, t_2)] \quad (3)$$

Because spatial variations of $a(x, y)$, $b(x, y)$ and $\phi(x, y, t)$ are slow compared with f_0 , the central lobe and the two side lobes in the frequency domain of the images $g(x, y, t)$ are separated, as illustrated in Fig. 3d. It is feasible to employ two-dimensional Fourier transform method to analyze the fringes. A band-pass filter centered at f_0 was applied to obtain one of the spectral side lobes of the images. For $g(x, y, t_1)$ and $g(x, y, t_2)$, the filtered images can be written in the following form,

$$g(x, y, t_1)' = \frac{1}{2} b(x, y) \exp i[2\pi f_0 x + \phi(x, y, t_1)] \quad (4)$$

$$g(x, y, t_2)' = \frac{1}{2} b(x, y) \exp i[2\pi f_0 x + \phi(x, y, t_2)] \quad (5)$$

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