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Analysis of charging-induced structural damage in electrochemical systems

Fuqian Yang

Electrochemical charging of an electrochemical system results in Maxwell stress and a variation in the surface energy of the electrode. The charging-induced variation in the surface energy of the electrode and Maxwell stress introduce mechanical deformation and create stresses in the electrode. To relax the strain energy stored in the electrode, surface cracking and buckling can occur. Under the condition that the charging-induced change in the surface energy is the dominant factor controlling the in-plane deformation of a planar electrode, *i.e.* the bonding between the adsorbate and the substrate is weak, the variation in the surface energy of the electrode can be described by the Lippmann equation. Using the Lippmann equation, both the surface charge density on the electrode and the surface energy of the electrode are formulated as a function of the electrode potential. Analytical relations between the critical electrode potential and average damage size have been obtained for the charging-induced cracking and buckling in a planar, thin-film electrode. The results show that surface cracking will prevail over local buckling in accordance with experimental observations. Both critical electrode potentials are inversely proportional to the square root of the magnitude of the differential capacity of the electrical double layer at the electrode potential of zero charge.

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1. Introduction

Electrochemical supercapacitors represent a class of energy-storage devices that convert electric energy into chemical energy in the form of an electrical double layer at the electrode–electrolyte interface. Most of the electrochemical supercapacitors are based on activated carbon as an electrode with a liquid electrolyte supported in a porous matrix (separator),¹ which is sandwiched between the electrodes. In general, it is assumed that there are no chemical changes and phase changes taking place within the electrodes or electrolyte during electrochemical charging and discharging,² which involve the motion of ions, *i.e.* ionic current, into and out of the electrodes.

It has been reported that carbon-based electrodes experienced volumetric changes during charging and discharging in both aqueous electrolyte solutions^{3–5} and ionic liquids.^{6–9} As suggested by Hahn,⁶ there are three possible mechanisms associated with the charging-induced dimensional change: (1) ion intercalation,^{6–11} (2) electron/hole injection,^{12,13} and (3) charge-regulated surface tension of the electrodes.^{3–5} The dimensional change induced by ion intercalation is generally controlled by the size difference between the molecule/atom of the host material and the solute molecule/atom or the size difference between the vacant site (interstitial site or lattice site) and the

solute molecule/atom; the dimensional change induced by electron/hole injection is dependent on the bond-length change associated with the charge transfer. The dimensional change induced by the charge-regulated surface tension of the electrode is due to the deformation dependence of materials on surface tension/stress.^{14–17}

There are reports on the deformation of the electrode associated with the charge-regulated surface tension of the electrode. Beck¹⁸ developed an extensometer to measure the variation in the surface tension/energy of an Au ribbon with an electric potential in 0.1 M KCl solution. Ibach *et al.*¹⁹ used an electrochemical STM (scanning tunneling microscope) to examine the bending of crystal plates for determining the potential-induced interface stress on Au(111) and Au(100) in 0.1 M HClO₄ and suggested that the Lippmann equation^{20,21} has little bearing on the potential-induced interface stress at the solid–liquid interface. Raiteri and Butt²² used the AFM (atomic force microscopy) cantilevers to measure surface-stress curves for gold and platinum from potential-induced bending of the cantilevers in aqueous electrolyte solutions. Bachmann and Miklavcic²³ analyzed the deformation of fluid interfaces by electrical double-layer forces. Kramer *et al.*²⁴ constructed a double layer structure consisting of an Au layer and an Au nanoporous layer and observed the tip displacements of several millimeters induced by the electrochemical potential in aqueous electrolytes. Haiss²⁵ has reviewed the adsorbate-induced changes in the surface stress tensor from cantilever bending experiments, and discussed the linear relation between surface

Materials Program, Department of Chemical and Materials Engineering,
University of Kentucky, Lexington, KY 40506, USA. E-mail: fyang2@uky.edu

stress and surface charge at the metal–electrolyte interface. All these have demonstrated the important role of the charge-regulated surface tension of the electrode in determining the dimensional change of the electrodes in electrochemical supercapacitors. Note that Haiss²⁵ pointed out that the terms associated with electrostriction are negligible and the thermodynamic analysis of solid electrodes with the focus on the surface energy can follow the same approach as liquid electrodes under normal experimental conditions.

While analyzing the nonlinear response of a metal surface to charging in interfacial electrochemistry, Feldman *et al.*²⁶ used an electromechanical model, which consists of a capacitor made of two plates with a plate linked to a spring, to analyze the interfacial relaxation in a metal/surface inactive electrolyte system. Using the same electromechanical model with a parallel capacitor as an electrical double layer, Partenskii and Jordan²⁷ analyzed the stability of charged interfaces under the control of charge density. Both analyses did not consider any structural damage potentially presented in the electrodes due to the variation in the surface energy induced by charging. Recognizing the possible effect of the charging-induced deformation on the structural durability of electrochemical supercapacitors, the strain energy due to the variation in the surface energy induced by charging is first incorporated in the analysis of a charged interface. The structural damage including surface cracking and buckling induced by the charge-regulated surface energy is then analyzed.

2. Physical model

The variation in the surface energy of a charged electrode in an electrolyte solution can be described by the electrocapillarity equation for an elastically stretched solid electrode²⁸ as

$$\frac{\partial \gamma}{\partial E} = -\rho + (g - \gamma) \frac{d\varepsilon}{dE} \quad (1)$$

for constant pressure, temperature and chemical potential. Here, γ is the surface energy of the electrode, whose properties are identical before and after the formation of a new surface, ρ is the surface charge density, E is the electrode potential, g is the surface stress, and ε is the surface strain. Note that the surface charge density is due to the formation of an electrical double layer near the charged electrode. The second term in eqn (1) is related to electrostrictive effect^{25,28} and it has been suggested that the contribution of the electrostrictive effect is negligible. Note that the surface stress can also be related to the deformation-induced formation of new surfaces.²⁹ In general, the change in the surface stress induced by adsorption will introduce the effects of charge transfer in electrolytes³⁰ and is dependent on the bonding strength between the adsorbate and the substrate (electrode). Recently, the slip boundary condition has been extensively used in the analysis of electro-osmotic flow.^{31–33} This trend likely suggests the weak bonding between the adsorbate and the substrate. For the weak bonding between the adsorbate and the substrate, such as a gold electrode in deaerated 1.0 mol dm³ NaClO₄ solution without iodide ions,^{34,35} the adsorbate-induced change in surface stress becomes negligible,

and $g \approx \gamma$. Under such a condition or the negligible effect of electrostriction, eqn (1) reduces to

$$\frac{\partial \gamma}{\partial E} = -\rho \quad (2)$$

From eqn (2), the differential capacity of the electrical double layer, C_d , can be calculated as³⁶

$$C_d = -\frac{\partial \rho}{\partial E} = \frac{\partial^2 \gamma}{\partial E^2} \quad (3)$$

and the integral capacity of the electrical double layer, C_i , as³⁶

$$C_i = -\frac{\rho}{E} = \frac{1}{E} \frac{\partial \gamma}{\partial E} \quad (4)$$

It is evident that $\rho = 0$ at $E = 0$, which corresponds to the electrode potential of zero charge, E_{pzc} . Under the condition of constant composition, eqn (3) gives

$$\gamma = \gamma_0 + \int_0^E dE' \int_0^{E'} C_d(U) dU \quad (5)$$

where γ_0 is the surface energy of the electrode at $E = 0$. The Taylor series expansion of C_d around $E = 0$ is

$$C_d|_{E=0} = C_0 + C_1 E + O(E^2) \quad (6)$$

with $C_0 = C_d(0)$ and $C_1 = \partial C_d(0)/\partial E$. Substituting eqn (6) in eqn (5) yields

$$\gamma = \gamma_0 + \frac{1}{2} C_0 E^2 + O(E^3) \quad (7)$$

which reveals the field dependence of the surface energy of the electrode immersed in an electrolyte solution under the action of an electric field. Note that the sign of the quadratic term is different from that in the Lippmann equation²⁰ due to the use of eqn (3).

From eqn (1) and (7), the variation in the surface charge density with the electrode potential is found as

$$\rho = -\frac{\partial \gamma}{\partial E} = -C_0 E + O(E^2) \quad (8)$$

For a planar electrode in the form of a thin film of thickness h , as shown in Fig. 1, which has been extensively used in electrochemical supercapacitors and lithium-ion batteries, the charging-induced change in the surface energy will introduce in-plane deformation of the electrode, resulting in the evolution of in-plane stress due to the confinement of the substrate to the deformation of the electrode. Note that there also exists the adsorbate-induced change in surface stress, which can also cause the in-plane deformation of the electrode. As discussed above, the adsorbate-induced change in surface stress becomes negligible for the weak bonding between the adsorbate and the substrate. The present analysis is based on the condition that the charging-induced change in the surface energy is the dominant factor controlling the in-plane deformation of the electrode, *i.e.* the bonding between the adsorbate and the substrate is weak.

Assume that the planar electrode is supported by a stiff substrate, which confines the in-plane deformation of the electrode. The strain energy density, U_s , which is associated

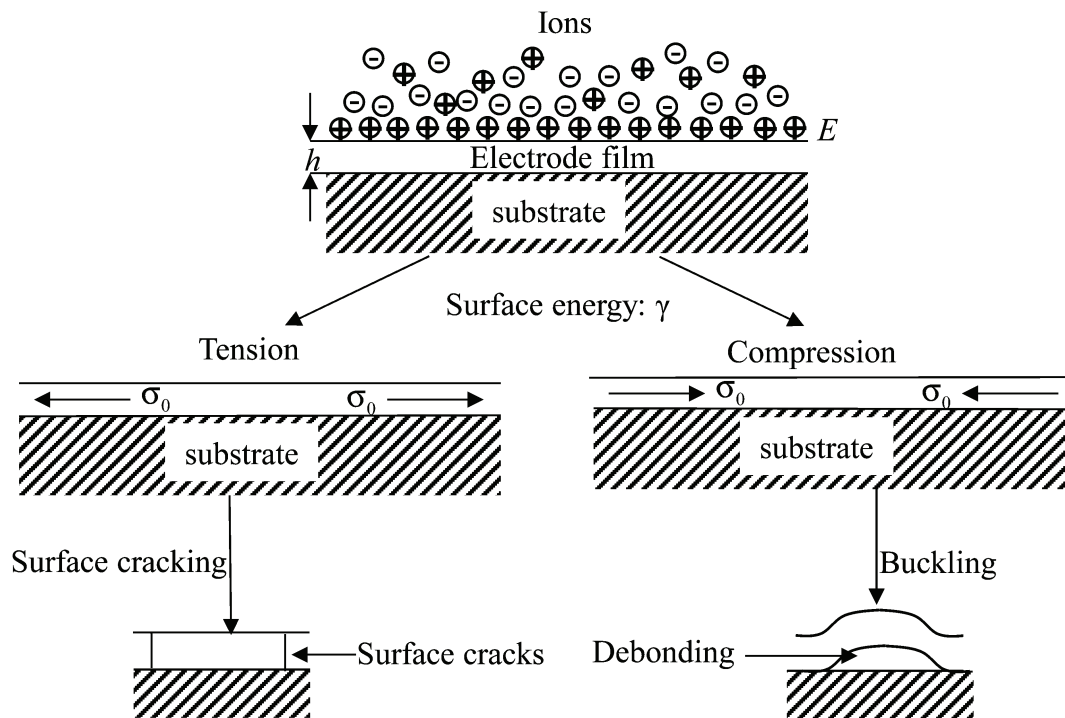


Fig. 1 Schematic diagram showing the structural damage caused by charging-induced change in the surface energy of an electrode film under the condition of weak bonding between the adsorbate and the substrate (σ_0 : stress).

with the charging-induced change of the surface energy, can be calculated as

$$U_s = \frac{1-\nu}{2Y} \left(\frac{\gamma - \gamma_0}{h} \right)^2 \approx \frac{1-\nu}{8Y} \frac{C_0^2 E^4}{h^2} \quad (9)$$

with Y and ν being the Young's modulus and Poisson's ratio of the electrode film, respectively.

It is known that the surface of an electrode with a surface charge density of ρ experiences normal Maxwell stress, σ_{Maxwell} , as

$$\sigma_{\text{Maxwell}} = \frac{1}{2} \frac{\rho^2}{\epsilon} \quad (10)$$

with ϵ being the dielectric constant of the electrolyte under the action of the electrostatic field. Substituting eqn (8) in eqn (10) yields

$$\sigma_{\text{Maxwell}} \approx \frac{C_0^2 E^2}{2\epsilon} + O(E^4) \quad (11)$$

The strain energy density associated with the deformation induced by the normal Maxwell stress, U_M , is

$$U_M = \frac{1}{2} \frac{\sigma_{\text{Maxwell}}^2}{Y} \approx \frac{1}{8} \frac{C_0^4 E^4}{Y \epsilon^2} \quad (12)$$

According to eqn (9) and (12), it is evident that both the strain energies are proportional to the square of C_0^2 and the fourth power of the electrode potential. However, the strain energy density associated with the charging-induced change in the surface energy, which is directly associated with the in-plane deformation of the electrode, is inversely proportional to the square of the film thickness and plays an important role in controlling the structural durability of the planar electrode. Thus, the deformation induced by the normal Maxwell stress

plays a negligible role in the structural durability of the planar electrode associated with the in-plane deformation, which is analyzed in the following section.

3. Results and discussion

From eqn (7), one can note that electric charging can lead to the increase or decrease of the surface energy of a charged electrode in an electrolyte solution, depending on the sign of C_0 . For positive C_0 , electric charging leads to the increase of the surface energy, and the electrode experiences compression; for negative C_0 , electric charging leads to the decrease of the surface energy, and the electrode experiences tension.

Surface cracking

As discussed above, the stress state of an electrode film is tensile if electric charging leads to the decrease of the surface energy. Subjected to tensile stress, surface cracking with the creation of two new surfaces can occur to release the stress (strain energy) introduced by electric charging. Assume that the charging-induced cracking is a slow process and leads to the formation of an array of parallel-through-thickness, straight cracks with an average distance of l_c between them. The decrease of the total energy due to the formation of an array of parallel-through-thickness, straight cracks requires

$$l_c h U_s \geq 2 h \gamma_1 \quad (13)$$

with γ_1 being the surface energy of the electrode film without any intimate contact with the electrolyte. Substituting eqn (9) in eqn (13) yields

$$E \geq 2 \left(\frac{Y}{1-\nu} \frac{\gamma_1}{l_c} \frac{h^2}{C_0^2} \right)^{1/4} \quad (14)$$

which gives the critical electrode potential for the formation of surface cracks, E_{cr} , as

$$E_{cr} = 2 \left(\frac{Y}{1-\nu} \frac{\gamma_1}{l_c} \frac{h^2}{C_0^2} \right)^{1/4} \quad (15)$$

The critical electrode potential is proportional to the square root of the film thickness and is inversely proportional to one fourth of the average distance between surface cracks. A large electrode potential will create dense surface cracks, which can readily cause structural damage to the electrode film during electric charging.

Surface buckling

It is known that the compressive stress acting on an elastic film can cause local buckling of the elastic film when the compressive stress in an elastic film reaches a critical stress.³⁷ Under the action that electric charging leads to the increase of the surface energy, the stress state of an electrode film is compressive, and local buckling can occur to release the stress (strain energy) introduced by electric charging and lead to the formation of two new surfaces from the film–substrate interface. From the principle of energy, the condition for the formation of the charging-induced buckling of an electrode film in a plate shape of size l_d is

$$l_d h U_s \geq l_d \Delta\gamma + l_d h U_B \quad (16)$$

with the Dupré constant of $\Delta\gamma$ as

$$\Delta\gamma = \gamma_1 + \gamma_2 - \gamma_{12} \quad (17)$$

Here, γ_2 is the surface energy of the substrate and γ_{12} is the interface energy for the film–substrate interface. The second term on the right side of (16) is the strain energy stored in the buckled plate after the delamination and buckling. The parameter of U_B is calculated from the result given by Gill and Rau³⁸ from the plate theory as

$$\begin{aligned} U_B &= \frac{\pi^2}{24} \left(\frac{h}{l_d} \right)^2 \frac{Y \varepsilon_{xx}}{1-\nu^2} = \frac{\pi^2}{24} \frac{1}{1-\nu^2} \left(\frac{h}{l_d} \right)^2 \left(\frac{\gamma - \gamma_0}{h} \right) \\ &= \frac{\pi^2}{24} \frac{1}{1-\nu^2} \left(\frac{h}{l_d} \right)^2 \left(\frac{C_0 E^2}{h} \right) \end{aligned} \quad (18)$$

with ε_{xx} being the in-plane strain induced by the charging-induced variation in the surface energy of the electrode. By substituting eqn (9) and (18) in eqn (16), one obtains

$$E^4 \geq \frac{8 Y h \Delta\gamma}{C_0^2 (1-\nu)} + \frac{\pi^2}{3} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{C_0 (1-\nu)} \frac{E^2}{1-\nu^2} \quad (19)$$

which gives

$$\begin{aligned} E^2 &\geq \frac{\pi^2}{6} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{C_0 (1-\nu)} \frac{1}{1-\nu^2} \\ &+ \frac{1}{C_0} \left[\left(\frac{\pi^2}{6} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{(1-\nu)} \frac{1}{1-\nu^2} \right)^2 + \frac{8 Y h \Delta\gamma}{1-\nu} \right]^{1/2} \end{aligned} \quad (20)$$

From eqn (20), the critical electrode potential for the initiation of the charging-induced buckling, $E_{cr}^{(b)}$, is found as

$$\begin{aligned} \left(E_{cr}^{(b)} \right)^2 &= \frac{\pi^2}{6} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{C_0 (1-\nu)} \frac{1}{1-\nu^2} \\ &+ \frac{1}{C_0} \left[\left(\frac{\pi^2}{6} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{(1-\nu)} \frac{1}{1-\nu^2} \right)^2 + \frac{8 Y h \Delta\gamma}{1-\nu} \right]^{1/2} \end{aligned} \quad (21)$$

The critical electrode potential is a function of the film thickness and the reciprocal of the delamination size. A large electrode potential is needed to cause local buckling/delamination of thick films with small delamination sizes. It is easy to introduce local delamination in planar electrodes of small thickness during electric charging. For $\Delta\gamma = 0$, eqn (21) gives

$$\left(E_{cr}^{(b)} \right)^2 = \frac{\pi^2}{3} \left(\frac{h}{l_d} \right)^2 \frac{h Y}{C_0 (1-\nu)} \frac{1}{1-\nu^2} \quad (22)$$

The critical electrode potential is proportional to the 3/2 power of the film thickness and inversely proportional to the delamination size.

It is worth mentioning that the above buckling analysis is based on that the surface of the film is smooth, as widely used in the mechanics of materials. For non-smooth surfaces with steps and multiple facets, one can follow the approach used by Müller *et al.*³⁹ in analyzing the discontinuities of surface stress on faceted surfaces to take into account the effects of faceted surfaces on the critical electrode potential.

According to eqn (15) and (21), both critical electrode potentials are inversely proportional to the square root of $|C_0|$, the magnitude of the differential capacity of the electrical double layer at $E = 0$. The differential capacity of the electrical double layer at $E = 0$ plays an important role in determining the critical electrode potentials controlling the mechanical degradation of the planar electrodes. It is worth pointing out that eqn (15) and (21) can also be used to measure the surface/interface energy from the charging-induced surface cracking or buckling.

It needs to be pointed out that the above analysis is based on the condition that the charging-induced change in the surface energy is the dominant factor controlling the in-plane deformation of the electrode, *i.e.* the bonding between the adsorbate and the substrate is weak. When the adsorbate-induced change in surface stress is not negligible, one needs to incorporate the contribution of the adsorbate-induced change in surface stress in the analysis of the structural damage of the electrode used in electrochemical supercapacitors and lithium-ion batteries.

Numerical calculations

Currently, most electrodes used in electrochemical supercapacitors are based on carbon-derived materials. Here, we consider a graphite-based electrode of 100 nm, which is coated on the surface of a cast iron (current collector) with an incoherent interface. For simplification, the graphite-based electrode is assumed to be isotropic and homogeneous. The elastic constants of graphite are $Y = 15.7$ GPa and $\nu = 0.23$.⁴⁰ The surface energies

of graphite and cast iron are $0.165 \text{ J m}^{-2,41}$ and $2.40 \text{ J m}^{-2,19}$, respectively. There are no data available for the interface energy of the planar C/Fe interface. The average value of the surface energy of graphite and the surface energy of cast iron is used as the interface energy of the C/Fe interface, *i.e.* $\gamma_{12} = 1.28 \text{ J m}^{-2}$, as an approximation.

Fig. 2 shows the dependence of the critical electrode potential on the average damage size of l (*i.e.* l_c or l_d). The critical electrode potential decreases nonlinearly with the increase of the average damage size. A small electrode potential will introduce less structural damage into the electrode than a large electrode potential. For the storage of the same energy, one needs to make compromise between the charging rate and the lifetime of electrochemical systems in order to limit any catastrophic, structural damage occurring during electrochemical cycling.

From Fig. 2, one can note that the critical electrode potential for the buckling/delamination approaches constant when the average damage size approaches infinity, *i.e.* there exists the smallest critical electrode potential below which no buckling/delamination will occur if there are no external loading and structural defects/degradation introduced by chemical reaction and processing. Letting $l_d \rightarrow \infty$ in eqn (21), one obtains the smallest critical electrode potential, $\text{Min}(E_{\text{cr}}^{(b)})$, as

$$\text{Min}(E_{\text{cr}}^{(b)}) = \frac{1}{C_0^{1/2}} \left[\frac{8Yh\Delta\gamma}{1-\nu} \right]^{1/4} \quad (23)$$

which is proportional to the $1/4$ power of the film thickness of the planar electrode and the Dupré constant.

According to Fig. 2, a smaller electrode potential is needed for surface cracking than that for surface buckling/delamination. This trend suggests that charging-induced surface cracking likely is a dominant factor for structural degradation of the electrode used in electrochemical systems. The observation of the surface cracking of films during electrochemical cycling for electrochemical supercapacitors^{42–44} supports this result. As reported by Haiss,²⁵ extremely high anodic potentials caused the loss of the adhesion of the gold film deposited on glass cantilevers with

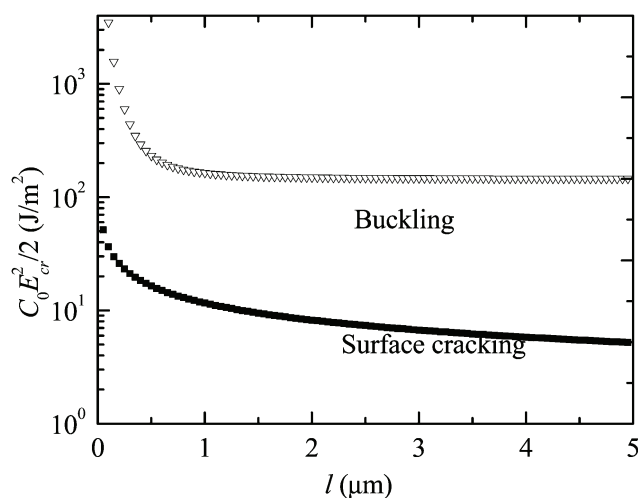


Fig. 2 Dependence of the critical electrode potential on average damage size.

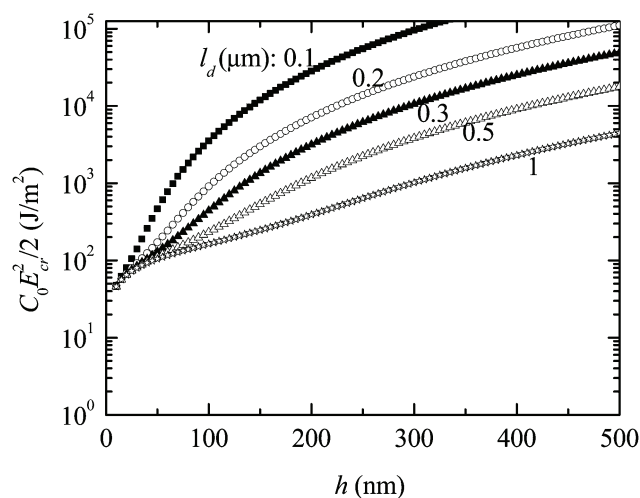


Fig. 3 Variation in the critical electrode potential with the film thickness for various buckling sizes.

a thin chromium film 2 nm in thickness between the gold film and the glass cantilevers for better adhesion. His result indirectly supports that a larger electrode potential is needed for surface buckling/delamination than for surface cracking.

Fig. 3 shows the variation in the critical electrode potential with the film thickness for various buckling sizes due to the charging-induced change in the surface energy of the planar electrode. The critical electrode potential increases with the increase of the film thickness for the same buckling/delamination size. A larger driving force is needed to produce the buckling/delamination of the same size in a thicker film. For thin films, local buckling/delamination leads to the release of most strain energy in the films through surface energy associated with the formation of new surfaces. For thick films, the formation of new surfaces cannot accommodate the strain energy stored in the films, and local buckling occurs to reduce the in-plane stress in the films. Electrodes made from ultrathin films have higher electrochemical durability than those made from thick films and should be used in electrochemical systems if structural durability is a major concern.

From eqn (15) and (21), one can note that the critical electrode potentials are dependent on Yh . It is known that the mechanical properties of low-dimensional structures are size-dependent. It is of practical importance to explore the size-dependence of the mechanical properties of low-dimensional structures in electrochemical environments.

4. Conclusions

The energy principle has played significant roles in the development of different disciplines, including thermodynamics, electrochemistry, and mechanics. It has become a useful tool in solving the problems of multi-fields. The effect of the interaction between an electric field and mechanical deformation in an electrochemical environment on structural degradation of the electrode and electrochemical systems cannot be simply described by a limiting process at the macroscopic level. The energy principle provides the foundation, *i.e.* the reduction of system energy, for the

development of the criterion in the analysis of the cracking and buckling behavior of materials due to the charging-induced variation in the surface energy of the electrode in electrochemical systems.

The work presented here has attempted to bring out the potential importance of the charging-induced deformation in the evolution of the structural degradation of the electrode during electrochemical cycling. Simple models have been developed to calculate the strain energies stored in a planar electrode on a rigid substrate (current collector) from the charging-induced variation in the surface energy of the electrode and Maxwell stress. The result reveals that the strain energy density associated with the deformation induced by the Maxwell stress is independent of the film thickness, and the strain energy density associated with the charging-induced change in the surface energy is inversely proportional to the square of the film thickness. For the surface cracking, the critical electrode potential is proportional to the square root of the film thickness and is inversely proportional to one fourth of the average distance between surface cracks. For the local buckling/delamination, the critical electrode potential is a function of the film thickness and the reciprocal of the delamination size. There exists the smallest critical electrode potential, below which no buckling/delamination will occur if there are no external loading and structural defects/degradation introduced by chemical reaction and processing. Both critical electrode potentials are inversely proportional to the square root of $|C_0|$, the magnitude of the differential capacity of the electrical double layer at $E = 0$.

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