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Review Article

How to minimise destabilising effect of gas bubbles on water splitting electrocatalysts?

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Summary

Development of efficient electrocatalytic gas-evolving electrodes is one of the essential prerequisites for the deployment of hydrogen-based electrochemical energy conversion and storage. Gas bubbles generated by electrolysis at electrocatalytic interfaces manifest into undesirable increase in overpotential that simultaneously compromises stability of the electrocatalytic materials. A key research question is how to use theory and advanced experimental tools to holistically understand the mechanism of gas-evolution phenomena and finally arrive at principles of electrode design that will assure facile gas evolution. The analysis given in this work offers an optimistic framework how to significantly reduce overpotential and enhance electrode stability during water electrolysis.

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Introduction

Electrochemical energy conversion is in continuous development [1]. The success of lithium-ion batteries is recognised globally [2]; however, a breakthrough in hydrogen energy is still missing [3]. After decades of solving challenges game changing progress remains elusive [4]. In the hydrogen conversion cycle, the initial

process is water electrolysis, whose efficiency besides thermodynamic voltage, is determined by energy dissipations including the following: kinetic overpotentials, ohmic drops, mass transport limitations and so on. During the last decade, the most intensive investigations were in the direction of electrocatalysis of the oxygen evolution reaction (OER). These efforts led to the design of exceptionally active electrocatalysts, which unfortunately routinely exhibited limitations during stability tests [5]. Systematic investigations of electrocatalyst stability initiated recently featured the central question: How to design active and stable electrochemical interfaces?

Learning from the past

To further improve water electrolysis, it is important to learn from well-established technologies and groundbreaking discoveries that led to major technological advancements. A great example is the introduction of dimensionally stable anodes (DSAs) for chlorine production in chlor-alkali technology in 1970s [6]. Reducing the voltage of the electrochemical reactor by more than 1 V [7], until today has had the consequence massive electricity savings worldwide. To understand this disruptive innovation, it is necessary to have technical data where the energy consumption of the electrochemical reactor is ‘dissected’ into components. Taking the example of the technical data given in [Table 1](#) that was analysed in detail in the past [7,8], rather than intrinsic electrocatalytic activity, the largest contribution to energy savings was minimisation of the gas bubble effect. The gas bubble effect is a consequence of a significantly higher concentration of the product than predicted by Henry’s law (supersaturation), at near-electrode region [9]. Gas bubbles block a fraction of the active sites [10] and enlarge the ohmic resistance of the electrolyte [11], which can have a dramatic impact on the reactor voltage [12]. The process of gas evolution comprises the following: nucleation, growth, coalescence and detachment [13]. Gas bubble detachment is the terminal step of gas evolution which makes previously covered active sites available again for reaction. Therefore, acceleration of gas bubble detachment is an essential process behind dramatic energy efficiency improvement of chlor-alkali technology.

Table 1

Comparison of voltage components, current efficiency and energy consumption of electrochemical reactor in chlor-alkali technology, for two different anodes, graphite and DSA^a.

	Graphite	DSA
Anode potential (V)	1.47	1.37
Cathode potential (V)	-1.85	-1.85
Anode ohmic drop (V)	0.15	0.15
Electrolyte ohmic drop (V)	0.60 ^a	0.40 ^b
Gas bubble effect (V)	0.90	0.13
Current efficiency (%)	96	97
Energy consumption (kWh t ⁻¹) ^c	3910	3040

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^a Anode-cathode distance 3 mm.

^b Anode-cathode distance 2 mm.

^c Current density 10 kA m⁻².

For water electrolysis there is apparently no available technical data similar to that shown in Table 1; nevertheless, the gas bubble effect must substantially contribute to voltage. Unfortunately, not many works were conclusive in trying to explain the mechanism(s) of fast bubble detachment in order to propose design principles for electrodes with superior properties [14], including the development of analytics to shed light on the complexity of gas evolution.

Mechanism of electrocatalytic gas bubble evolution

The first step in the mechanism is the nucleation process, governed by the critical radius that is proportional to the surface tension of the liquid and cosine of the contact angle of the liquid at the electrode surface as well as inversely proportional to the difference between external pressure and interfacial pressure. This should be straightforward from the Young-Laplace equation, although various literature sources are not coherent in their description of bubble nucleation [15,16]. Interesting is that the hydrogen evolution reaction (HER) that exhibits superior kinetics in comparison to OER, proceeds at unfavourable hydrophobic surfaces. Opposite is with OER that has severely inferior kinetics in comparison to HER, but proceeds at desirable hydrophilic surfaces. At the same time, gas bubbles in electrolytes with pH < 2 carry positive charge, while those with pH > 3 carry negative charge. Therefore, negatively charged HER cathode will additionally attract bubbles in electrolytes with pH < 2 and repulse bubbles in electrolytes with pH > 3. Analogy can be drawn for OER anodes [17]. Failure to separate the impact of intrinsic activity from the physics of gas evolution can introduce uncertainty in catalytic trends [18]. Therefore, gas evolution exhibits a strong dependence

on the hydrophilicity of the electrode surface and also on electrolyte pH [8,19].

After nucleation, gas bubbles grow proportionally to the applied overpotential, number of active sites, distribution of active sites and so on [20]. Finally, after growth and certain period of time spent at the electrode surface, the gas bubble is getting detached [21]. To understand the average period that one gas bubble spends on the electrode surface and effectively blocks active sites, it is instructive to recall the analogy between phenomena of gas evolution and physics of boiling [22], despite this analogy having limitations [23]. In the physics of boiling, it is well established that the radius of gas bubble detachment is inversely related to the detachment frequency. In other words, if one can keep the gas bubble radius small, the frequency of detachment should be higher than for larger bubbles. This is a consequence of the mechanism of bubble detachment. Namely, gas bubbles are, due to significant amount of thermal energy, in constant oscillatory motion. Oscillatory motion of bubbles causes opposing flows of the liquid towards the root of the bubble. The smaller the gas bubbles are, the more frequent are the opposing flows of the liquid that can ultimately cause bubble detachment.

Electrode morphology design

To accelerate gas evolution, one has to limit the growth and coalescence in confined regions of the electrode surface. If the interfacial layer would be observed statically or 'frozen', then the surface covered by larger bubbles would have more available active sites between bubbles, than in the case of small bubbles. However, the system is dynamic meaning that the interface with smaller bubbles and higher detachment frequencies manifest overall higher accessibility of active sites [18]. A schematic of the gas evolution process is visualised in Figure 1 at a DSA-type of surface [24].

Gas bubbles nucleated in 'cracks' are limited in growth and they detach frequently with relatively small radii. To assure stable nucleation and to minimise growth, 'cracks' should have adequate depth, because the critical overpotential for gas bubble nucleation drops with increase in thickness of the catalyst layer [25]. Growth of gas bubbles is possible at the outer surface of 'islands', but due to frequent micro-convection into the 'cracks', growth at the outer surface of the 'islands' will be interrupted. Therefore, the electrode surface has to be with defined 'crack' size and defined thickness of the catalyst layer. This is possible by controlling the tensile stress during synthesis [24]. The efficiency of gas evolution is essentially an interplay of surface morphology and operational parameters [26]. This has a consequence on a very important property for kinetic studies known as the effective surface area (A_{eff}), which reflects

Figure 1

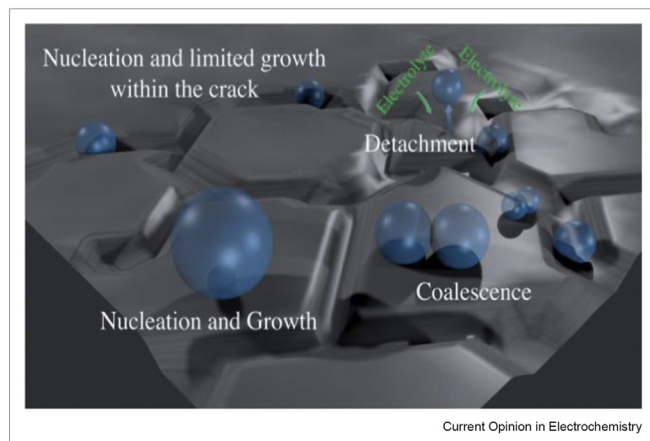


Illustration of gas evolution mechanism on an electrode with an internal hydraulic regime. Reprinted from Zeradjanin et al. [24] - Published by The Royal Society of Chemistry.

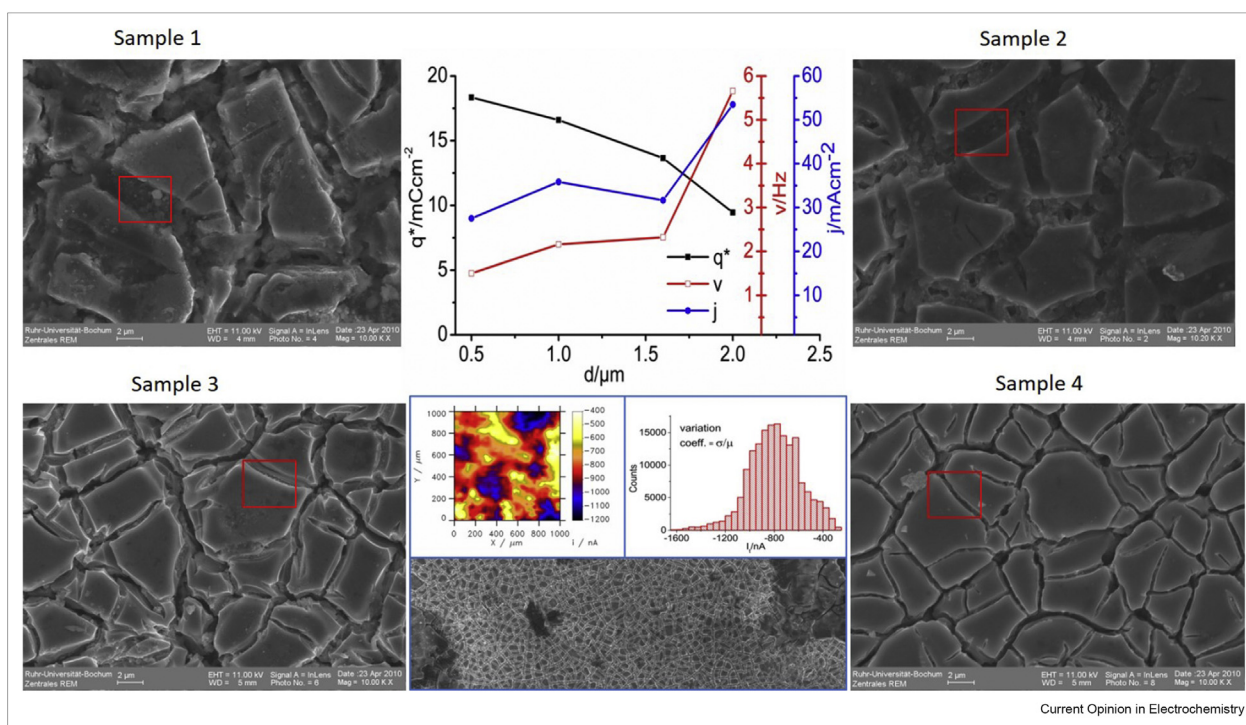
the number of active sites that are not covered by gas bubbles and quantitatively is between geometric area (A_{geo}) and total surface area [18]. In the past it was shown that the extremely high pressure (i.e., hundreds

of bars) was necessary to activate nanopores to participate in the reaction [27]. Pores will be filled with electrolyte if the pressure difference is high enough to overcome surface tension of the liquid. At room temperature conditions, it seems that pores of relevance should preferentially be on a microscale rather than nanoscale [28]. At the same time, there are numerous efforts in designing morphology on nanoscale [29–36].

Analytical tools and spatiotemporal characterisation

For four DSA samples with almost identical chemistry, electrochemical properties are analysed as function of geometric properties (i.e., ‘crack’ size) as shown in Figure 2. Although voltammetric surface charge (analogue to number of active sites) was steadily dropping with increase in ‘crack’ size, apparent turnover (current density normalised by voltammetric surface charge) was increasing steadily up to ‘crack’ size of approximately 1.5–2.0 μm and then turnover rapidly increased almost 300%. Considering that DSA samples had almost identical chemistry, the drastic enhancement in performance was attributed to significantly accelerated gas evolution. As stated previously, facile gas evolution happens when gas bubbles are limited in growth. Modelling also indicates that nucleation-driven

Figure 2



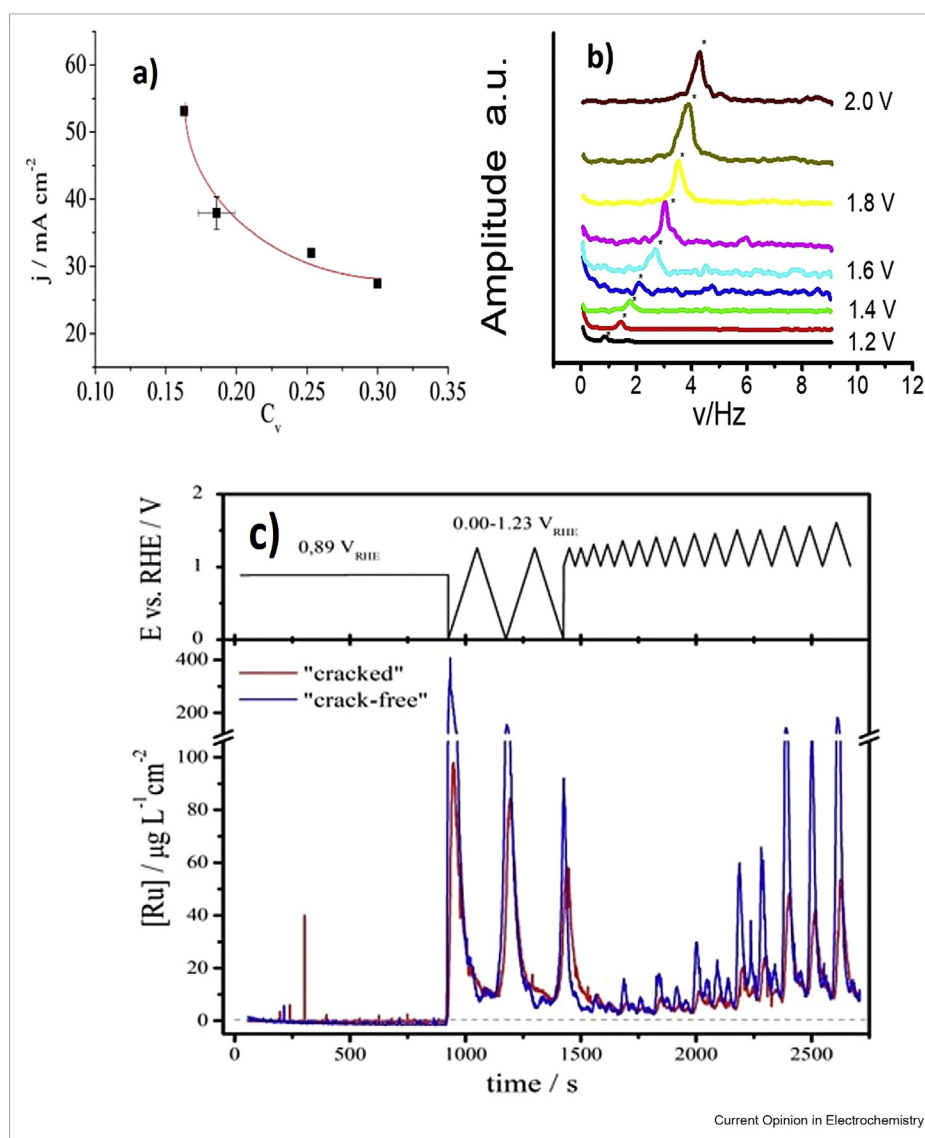
Current density (j), voltammetric surface charge (q^*) and apparent turnover frequency (Hz) of the four DSA samples with different average ‘crack’ size (d), as indicated with red rectangular in corresponding SEM micrographs. Reprinted from Zeradjanin et al. [28] with permission from Elsevier. Below the correlation between electrochemical and geometric properties is an illustration of the spatial distribution of activity as SECM scan and corresponding histogram, including SEM of scanned inhomogeneous DSA surface. Reprinted from Zeradjanin et al. [8] with permission from Elsevier. SEM, scanning electron microscopy; SECM, scanning electrochemical microscopy.

bubble detachment could result in the lowest overpotentials [37]. Besides morphology, of major importance is spatial distribution of the morphological pattern. Figure 2 (centre/down) shows that local catalytic current can vary significantly, due to morphological heterogeneities, as evaluated by scanning electron microscopy and scanning electrochemical microscopy (SECM).

Local current distribution can be expressed by variation coefficient, that is, the standard deviation of local current normalised by mean local current. Figure 3a shows that more uniform current distribution results in better overall performance, which is an important fact for the

electrode preparation process. Arguably, the most important aspect of characterisation is to monitor frequencies of gas bubble detachment. Detailed analysis of potential-dependent frequency spectra (Figure 3b) recorded by SECM was given previously [18]. In general terms, one can analyse how gas bubble detachment frequencies develop with overpotential. If frequency changes easily with overpotential, bubble growth is kinetically controlled and concentration overpotentials are minimised. Also, if gas bubbles evolve with one single frequency, this indicates preferential radius of detachment. From empirical data, electrodes with uniform gas bubble detachment frequency are generally known to have excellent performance in comparison to

Figure 3



Spatiotemporal oscillations and electrode stability. (a) Overall current density as function of variation coefficient as indicator of local current distribution obtained by SECM. Reprinted from Zeradjanin et al. [8] with permission from Elsevier. (b) Potential-dependent frequency spectra obtained by SECM. Reprinted from Zeradjanin et al. [18] with permission from Wiley. (c) Time-resolved potential-dependent dissolution profile obtained by electrochemical cell coupled to inductively coupled plasma mass spectrometer. Reprinted from Zeradjanin [24] - Published by The Royal Society of Chemistry.

those with multiple detachment frequencies [26]. For the future, it is very important to conduct high-resolution spatiotemporal analysis of electrocatalytic performance [38] including analysis of time-dependent frequency spectra [39].

In industrially relevant galvanostatic electrolysis, for a predefined mass of product proportional to current intensity, current density as well as overpotential will be lowest for the electrode with highest number of accessible active sites. That will be the case for the electrode with very facile gas evolution, meaning high effective surface area. If the effective surface area is low, local high current density causes high local overpotential that induces severe local catalyst dissolution during OER, thus revealing an enormous impact on stability of electrodes [40]. *In situ* time-resolved experiments on catalyst dissolution during OER with intensive gas bubble evolution on various morphologies, especially in the galvanostatic regime, are to our knowledge not reported in literature.

Figure 3c shows potentiodynamic perturbation and dissolution profile for samples with almost identical chemistry, but different morphology. A ‘cracked’ sample that at 1.45 V exhibited two times higher current density at the same time exhibited two times lower dissolution in comparison to a ‘crack’-free sample [24]. Accelerated gas evolution on the ‘cracked’ sample caused more intensive natural convection of electrolyte while in the case of the ‘crack-free’ sample highly acidified electrolyte remains in the inter-bubble spacing and allows for accelerated corrosion [24]. This is a clear case how morphology design can be the way to reduce overpotential and enhance stability of electrocatalysts under aggressive operational conditions.

Because of a negligible impact of nanostructuring on overpotential in our experiments, we focused on the microscale. However, nanobubbles exist at the electrode surface and play a role that is still not resolved [41]. Existing reports are contradictory stating that nanobubbles grow into microbubbles before getting detached from the surface [13] or completely the opposite, claiming that nanobubbles do not grow but dissolve in the electrolyte [42]. Interesting for reader could be analysis where nanobubbles were investigated using nanoelectrodes [43]. Besides nanoelectrodes being interesting tools to study nanobubbles, an interesting tool for studying nanobubble in parallel with microbubbles would be the electrochemical quartz crystal microbalance [44]. At the end, worth to mention are approaches to accelerate gas evolution externally using centrifugal field, ultrasonic field, magnetohydrodynamic field and so on [45], or using surfactants [17,46–48]. The role of magnetic field was recently re-examined [49] revealing tremendous impact on gas evolution [50].

Conclusions and perspectives

This comprehensive perspective to understanding gas bubble evolution comprises the following: (1) hypothesis on mechanism, (2) experimental tools for spatiotemporal analysis of activity and *in situ* analysis of stability and (3) morphology design was shown as an example how to acquire knowledge and use it to improve existing state-of-the-art electrodes. Importantly, it was shown that a carefully designed morphological pattern can be a pathway to simultaneously reduce overpotential and enhance stability of electrodes under operational conditions. With our approach we hope to encourage future creative endeavours, especially those attempting to resolve behaviour and impact of nanobubbles.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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