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Electrocatalytic activity and anodic stability of electrodeposited ruthenium-rhodium coatings on titanium

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The stabilizing effect of rhodium addition to ruthenium coatings on titanium under anodic polarization in acidic solutions is described. The addition of rhodium to the coating leads to both an overall improved stability of the electrode, and an increase of the electrochemically active surface area. The service-life of a Rh-Ru coating polarized at 0.12 A cm^{-2} in 0.5 mol dm^{-3} sulphuric acid is improved by at least two orders of magnitude, while deterioration of the electroactive surface area never exceeds 30%. The rotating ring-disc electrode measurements show the dissolution of the coating at potentials above the onset of the oxygen evolution reaction at 1.1 V versus SCE.

1. Introduction

In electrolytic hydrogen production, as a basis for clean technologies, the choice of electrode materials is one of the most important issues in meeting optimal working conditions, both from the economical and environmental point of view [1]. The kinetics of reaction on the oxygen electrode, being the slowest step in the water electrolysis, determines the electrocatalytic efficiency of the overall process. In this connection, ruthenium, as an anodic material in the oxygen evolution reaction, exhibits excellent electrocatalytic properties [2]. Unfortunately, during this process, it forms soluble intermediates with oxygen [3], and, therefore, is not useful for long-term applications. A number of procedures have been tested for the sake of improving its long-term stability. Many of them are based on modified Ru thin films deposited on various metallic substrates. Modifications of the films included the co-deposition of iridium by reactive sputtering [4, 5], the thermal decomposition of the mixture of ruthenium and iridium salts on titanium and formation of their oxides [6, 7] and the formation of mixed Ru-Ir coatings by electrodeposition of ruthenium and iridium salts on titanium [8, 9]. In a comparative study of anodic behaviour of electrodeposited Ru, Ir and Rh coatings on titanium, we have shown [10] that rhodium exhibited the best stability in the oxygen evolution reaction. It is the aim of the present paper to determine whether an addition of rhodium may preserve good electrocatalytic properties of a ruthenium electrode during a long-term operation.

In this work, titanium is chosen as a substrate for two reasons: it is highly resistive material on which ruthenium [11] and mixed ruthenium-iridium coatings have already been successfully electrodeposited [8], and, as a cheap material presents a good basis for commercial electrodes. It has also been used as a substrate for DSA anodes, obtained by thermal decomposition of $RuCl_3$ [12], in the chlor-alkaly industry for several decades.

2. Experimental procedure

The substrates for electrodeposition were titanium wires (Goodfellow) of 0.25 cm^2 geometrical area and plates (Goodfellow) ($10 \times 10 \times 0.5$ mm^3) fixed, in both cases, by epoxy resin into a glass tube, and titanium rods (Goodfellow) mounted in a rotating ring-disc electrode (RRDE) assembly. The titanium was polished with emery paper, and 1 μm alumina powder, washed and treated potentiodynamically from −0.25 V to 1.25 V versus saturated calomel electrode (SCE) in 0.5 mol dm^{-3} H_2SO_4 for 2 min in order to remove adsorbed organic impurities. The electrodeposition was carried out galvanostatically at 40 mA cm^{-2} current density for 15 min at 25 °C. Various contents of metal components in the coating were achieved by varying the concentration of diammonium hexachloro salts of Ru(IV) and Rh(IV) (Fluka, puriss. p.a.) in the plating bath while keeping the total concentration of metal salts constant, 1 g dm^{-3} in 1 mol dm^{-3} HCl.

The samples prepared for the X-ray photoemission spectroscopy (XPS) studies were washed with distilled

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TABLE I Composition of the plating baths and composition of the coatings obtained by XPS

Coating	Concentration of metal salts in the plating bath		Composition as determined by XPS	
	$(NH_4)_2RuCl_6$ g dm^{-3}	$(NH_4)_2RhCl_6$ g dm^{-3}	%Ru	%Rh
$Ru_{0.7}Rh_{0.3}$	0.8	0.2	73 ± 3	27 ± 3
$Ru_{0.3}Rh_{0.7}$	0.5	0.5	33 ± 3	67 ± 3
$Ru_{0.2}Rh_{0.8}$	0.2	0.8	23 ± 3	77 ± 3

water, dried, and transferred immediately into the ultrahigh vacuum (UHV) of an XPS apparatus. The radiation source was a Mg/Al dual-anode, operating at an energy of 1253.6 eV (MgKα line). The spectra were recorded using a 180° hemispherical analyzer (Vacuum Science Workshop HA 100). As the only task of the XPS analysis was to determine the Ru/Rh ratio of the coating, the following procedure was established: The spectra were recorded from "as received" samples and after each of several successive Ar^+ sputterings (altogether several minutes at 500 eV ion energy and 1–2 μA ion current) performed solely in order to remove some of the adsorbed contamination and thus improve the Ru and Rh signal intensities. Sputtering induced some changes (as expected) in the line shape of the spectra but did not influence the Ru/Rh ratio. As the Ru $3d_{3/2}$ peak overlaps with the C 1s peak, the elemental concentration of both constituents was judged from the peak area of their $3d_{5/2}$ peaks. The peak area integration was performed after a Shirley background subtraction, and care was taken to integrate the overall contributions to the peak. The values obtained were then corrected for the corresponding empirical sensitivity factors [13]. Table I shows the composition of plating baths used for Ru-Rh coating production, the composition of a coating as determined by the XPS, and the label of the sample used throughout this paper.

Electrodeposition, cyclic voltammetric characterization of coatings and anodic stability measurements were carried out using a potentiostat/galvanostat (EG&G Model 273/97). The RRDE measurements were carried out using a bipotentiostat (PINE Model AFRDE4E) controlled by a rotator (PINE Model AFMSRXE). In the rotating ring-disc electrode (PINE Model AFMT135PTPTT) the removable platinum disc was replaced with titanium. The ring electrode was made of platinum. The collection efficiency of the RRDE system reported elsewhere [14] was 0.20. The electrode was rotated at 2500 rotations per minute (r.p.m.).

All experiments were carried out at room temperature.

3. Results

3.1. Surface/electrochemical characterization of the coatings

Here we present data obtained for pure ruthenium and rhodium coatings on titanium together with data for three different Ru-Rh coatings, selected for their characteristic behaviour.

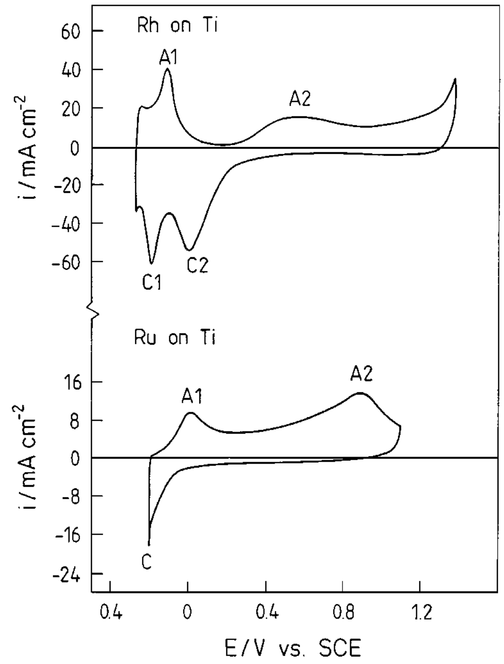

Figure 1 Cyclic voltammograms at a sweep rate of 50 mV s^{-1} in 0.5 mol dm^{-3} H_2SO_4 of electrodeposited rhodium and ruthenium coatings on titanium.

Fig. 1 shows the cyclic voltammograms of pure ruthenium and rhodium coatings on titanium in 0.5 mol dm^{-3} H_2SO_4. As far as their surface electrochemical oxidation/reduction processes are concerned they exhibit, together with other noble metals, some common characteristics, but they also exhibit several important differences. After hydrogen ionization peaks (A1) in the cathodic direction, the surface oxidation commences (A2 peaks). In the case of ruthenium, it starts immediately after hydrogen ionization. In fact, at the very beginning of the oxide formation (at 0.1 V), these two processes overlap. In the case of rhodium, a short but distinguishable double layer region exists at about 0.1 V. Cathodic processes are even more different. In the case of ruthenium only one (C) peak appears, where the majority of oxide is reduced in the hydrogen evolution region. Cathodic processes of rhodium give rise to two well resolved peaks, which are due to the oxide reduction (C2 peak) and deposition of hydrogen (reversible C1 peak).

The cyclic voltammograms of the three Ru-Rh coatings are shown in Fig. 2. By comparing these data with those in Fig. 1, it is evident that the $Ru_{0.7}Rh_{0.3}$ coating exhibits the Ru-type behaviour, the $Ru_{0.2}Rh_{0.8}$ coating exhibits the Rh-type behaviour while a mixed behaviour is obtained for the $Ru_{0.3}Rh_{0.7}$ coating.

3.2. Electrocatalytic efficiency of the coatings

The quasi-stationary potentiostatic polarization curves for the oxygen evolution reaction in 0.5 mol dm^{-3} H_2SO_4 of pure ruthenium, $Ru_{0.7}Rh_{0.3}$ and $Ru_{0.3}Rh_{0.7}$ coatings are shown in Fig. 3. A sharp decrease of a current above 1.32 V, in the case of pure ruthenium coating, was an indication of the dissolution of the ruthenium layer. Namely, it is known from previous

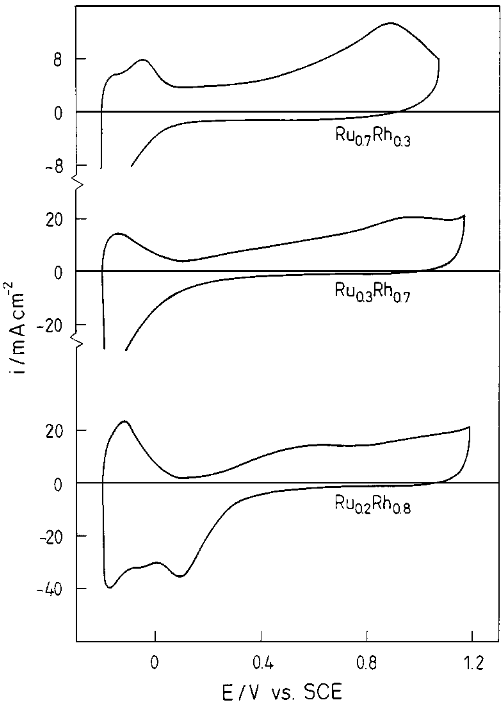

Figure 2 Cyclic voltammograms at a sweep rate of 50 mV s^{-1} in 0.5 mol dm^{-3} H_2SO_4 of the coatings on titanium.

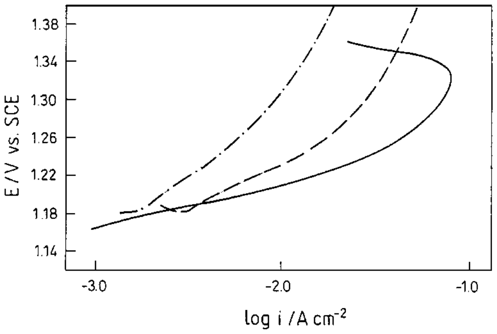

Figure 3 Quasi-stationary potentiostatic polarization curves at 1 mV s^{-1} in 0.5 mol dm^{-3} H_2SO_4 of (———) ruthenium, (- - - - -) $Ru_{0.7}Rh_{0.3}$ and (· - - - - -) $Ru_{0.3}Rh_{0.7}$ coatings on titanium.

TABLE II Ring currents (Fig. 4) normalized to a real surface area

Coating	iR/μA	Charge of oxide formation/mC	Real surface area cm^{-2}	$i_{norm}/\mu A\ cm^{-2}$
$Ru_{0.7}Rh_{0.3}$	10	36	43	0.23
$Ru_{0.3}Rh_{0.7}$	16	54	64	0.25
$Ru_{0.2}Rh_{0.8}$	18	71	85	0.21

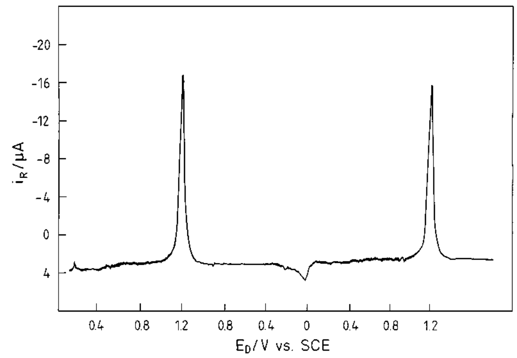

Figure 4 Currents at a Pt-ring electrode held at 0.8 V during two successive potentiodynamic sweeps from 0 to 1.2 V at a sweep rate of 50 mV s^{-1} in 0.5 mol dm^{-3} H_2SO_4 of electrodeposited $Ru_{0.3}Rh_{0.7}$ coating on a titanium disc electrode. Rotation speed 2500 r.p.m.

measurements [11] that this is not a passivation process. In the case of porous electrodes, like those obtained by electrodeposition, the electrochemical active surface area strongly depends on the thickness of a coating. Therefore, a decrese in electroactive surface area leads to a decrease in oxygen evolution current. Fig. 3 shows that the addition of rhodium is accompanied by a decrease in the electrocatalytic activity, on the one hand, and by an increase in the stability, on the other hand. Namely, there is an absence of the current decrease in the case of the mixed Ru-Rh coatings.

3.3. Stability measurements by rotating ring-disc electrode

The RRDE is a convenient tool for detection of dissolved species from a disc electrode provided that intermediates react electrochemically on the ring electrode [15]. Fig. 4 shows RRDE measurements for the $Ru_{0.3}Rh_{0.7}$ sample were potentiodynamically polarized during two successive potential sweeps from 0 V to 1.2 V, i.e., between the potentials of hydrogen evolution and oxygen evolution reactions (the other coatings show a similar behaviour; see Table II). The current maxima are observed at 1.2 V (Fig. 4), and inspection of Fig. 3 shows that, indeed, this potential is in the range of the oxygen evolution reaction. The potential of the ring electrode is held at 0.6 V in order to avoid the interferrence of oxygen reduction current which occurs at more negative potentials. The electrodeposited layer of $Ru_{0.3}Rh_{0.7}$ coating on titanium is not completely stable in the entire range of potential during the potential cycling. There is a small residual positive current of about 0.4 μA until the potential reaches 0 V when hydrogen evolved at the disc electrode is transferred and oxidized at the ring electrode. However, the dissolution current is small compared to the current of oxide formation (cyclic voltammograms in Fig. 2). The ring current is in the microampere range even while taking into account the value of 0.2 of the collection efficiency [14].

3.4. Determination of electrochemically active surface area

The peaks of ring currents at 1.2 V in Fig. 4 were normalized to the electrochemically active surface area. The charge of hydrogen ionization and/or deposition, which is usually used in real surface area determination of platinum electrode [16] was avoided here due to complications induced by the overlapping of hydrogen ionization and oxide formation, as well as by absorption of hydrogen in ruthenium [17]. Instead, the charge of oxide formation was used. According to the literature on anodically polarized ruthenium and rhodium electrodes, the formation of RuO_2 [18] and Rh_2O_3 [19] takes place before oxygen evolution. Therefore, the formation of those species is assumed also for the mixed

TABLE III Service-life and overvoltage at 0.12 A cm^{-2} of ruthenium and mixed Ru-Rh coatings on titanium

Coating	Service-life min	Overvoltage* η/V
Ru	1.5	1.25
$Ru_{0.7}Rh_{0.3}$	20	1.50
$Ru_{0.3}Rh_{0.7}$	240	1.55
$Ru_{0.2}Rh_{0.8}$	1200	1.75

*Measured at the beginning of the polarization.

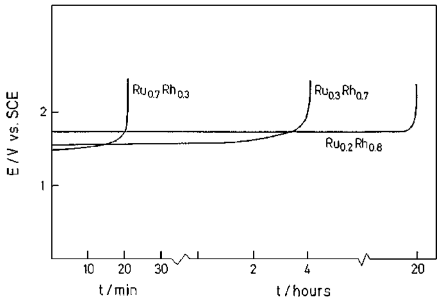

Figure 5 Potential-time curves during anodic galvanostatic polarization at 0.12 A cm^{-2} in 0.5 mol dm^{-3} H_2SO_4 of $Ru_{0.7}Rh_{0.3}$, $Ru_{0.3}Rh_{0.7}$ and $Ru_{0.2}Rh_{0.8}$ coatings on titanium.

coatings. In that case, the fractional number of electrons between 3 and 4 is calculated according to the composition of a coating. A charge of 420 μC per cm^2 is usually used for formation of oxide monolayer in the case of two-electron reaction on noble metals (formation of PtO, for example) [16]. Data, normalized to the electrochemically active surface area and summarized in Table II, show a decrease in electrode dissolution with the increase in rhodium content.

3.5. Stability measurements by anodic galvanostatic polarization

Another stability test was carried out using galvanostatic polarization at 0.12 A cm^{-2} in 0.5 mol dm^{-3} H_2SO_4 where a sudden increase in potential (Fig. 5) was a consequence of the exposure of the titanium surface to the solution induced by dissolution of the coating. The most stable was the $Ru_{0.2}Rh_{0.8}$ coating which, after six hours of anodic galvanostatic polarization, did not exhibit a change in potential, which would indicate a complete dissolution of the electrode layer (Table III). The coating exhibited about 30% decrease in the voltammetric charge of both the hydrogen ionization and deposition and of the oxide formation (Fig. 6). The service life of this electrode was 20 hours (Fig. 5).

4. Discussion

The purpose of modification of electrocatalytically active ruthenium surface with addition of stabilizing atoms is to obtain optimal conditions in a trade-off between activity and stability. In other words, to obtain optimal conditions by slowing down the high rates of oxygen evolution and ruthenium dissolution. Several successful attempts have been reported using codeposition of ruthenium with iridium [4–9], zirconium [20], antinomy [21] and cerium [22]. This is an alternative approach to thermal treatment of electrodeposited thin films of ruthenium [11, 23] and formation of RuO_2, in an analogy to DSA production of commercial anodes [12].

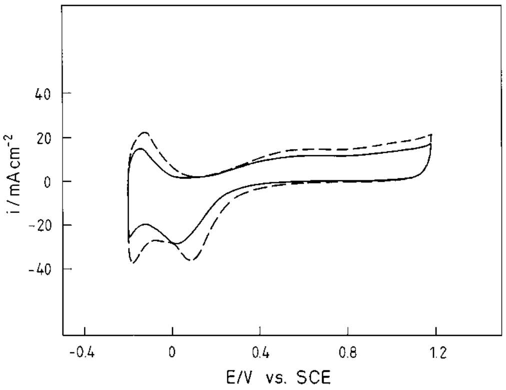

Figure 6 Cyclic voltammograms at a sweep rate of 50 mVs^{-1} in 0.5 mol dm^{-3} H_2SO_4 of $Ru_{0.2}Rh_{0.8}$ coating on titanium (———) before, and (- - - - - -) after 6 hours of anodic galvanostatic polarization at 0.12 a cm^{-2} in 0.5 mol dm^{-3} H_2SO_4.

The oxygen evolution reaction on noble metals occurs on oxide covered surfaces via a complex sequence of electrochemical and chemical reactions. They involve electron transfer and recombination of adsorbed intermediates usually written as:

$$S + H_2O \rightarrow SOH + H^+ + e^- \qquad (1)$$

$$2\,SOH \rightarrow SO + S + H_2O \qquad (2)$$

$$2\,SO \rightarrow 2S + O_2 \qquad (3)$$

where S stands for an active metal oxide site. For example, in the case of ruthenium, after formation of a RuO_2 layer at 1.1 V, at more positive potentials the oxidation is extended [24] to ruthenium in (+6) oxidation state (S in step 1). This species is in one-electron transfer reaction oxidized to (+7) state (step 1), followed by a disproportionation reaction and formation of ruthenium in its (+8) and (+6) states, respectively (step 2). The reaction sequence ends with the release of oxygen from ruthenium (+8) state (step 3). This is RuO_4 (or hydrated species, $RuO_3(OH)_2$ for example). Such a species is unstable and dissolves from the surface layer in either gaseous RuO_4 form or dissolved H_2RuO_5.

The high electrocatalytic activity of ruthenium in the oxygen evolution reaction, accompanied by the dissolution of ruthenium, is demonstrated in Fig. 3. Three anodic potentiostatic polarization plots are shown for pure ruthenium, for $Ru_{0.7}Rh_{0.3}$ and for $Ru_{0.3}Rh_{0.7}$, respectively. Ruthenium exhibits the lowest Tafel slope (30 mV/decade) and the highest current density at the same potential, which after 1.32 V decreases due to the above mentioned dissolution of surface layer and consequently, due to decrease in the real surface area. It should be pointed out that a potential of current maximum in Fig. 3 is not fixed at 1.32 V. It depends on the rate of anodic polarization, and shifts towards lower potentials with slower speed of anodic polarization. The increase in the current density exhibited by pure ruthenium coating is, compared to the mixed Ru-Rh coatings, not due to the increase of the real surface area. Moreover, the electrochemically active surface area, due to higher current efficiencies of rhodium electroplating, increases with the addition of rhodium (Table II). Therefore, the increase of current in the case of ruthenium is purely electrocatalytic.

The difference in Tafel slopes originates from differences in reaction mechanisms and rate determining steps. According to Bockris [25] when step 2 is rate determining, and this is the case with ruthenium reported previously [11, 26] as well as in this work, the Tafel slope is 30 mV/decade. When step 1 is rate determining, the corresponding Tafel slope is 120 mV/decade. This is the case with rhodium in an acid solution, and as far as the Tafel slope is concerned, $Ru_{0.3}Rh_{0.7}$ exhibits rhodium-like behaviour.

What is of particular interest for the present study is to what an extent the addition of rhodium stabilizes the ruthenium coating. This effect was examined firstly, under potential cycling conditions in the potential range between hydrogen and oxygen evolution reactions and secondly, at high positive potentials in the region of the oxygen evolution reaction. The stabilization effect under potential cycling conditions was determined from the height of the ring current at 1.2 V at various Ru-Rh coatings.

The increase in the ring current (Fig. 4) with the increase of the rhodium content in the coating implies an enhanced electrode dissolution. The analysis presented in Section 3.3 and the results shown in Table I show that a relationship exists between ring currents of metal dissolution and the increase in the electrochemically active surface area expressed by the increase of the voltammetric charge. The current decreases to 0.21 μA cm^{-2} (real surface area) at the $Ru_{0.2}Rh_{0.8}$ coating. Taking into account the value of 0.20 of the collection efficiency of the RRDE system used [14] the dissolution current at the disc electrode is 1.05 μA cm^{-2}. This value is in very good agreement with the value of 1.16 μA cm^{-2}, reported by Sverdlova *et al.* [27] for dissolution of rhodium electrode under potential cycling conditions at 54 m V s^{-1}. The dissolution of other noble metals, including ruthenium, during potential cycling in acid solution is well known [28]. The dissolution rate increases from platinum to ruthenium.

The stability of the coating, by addition of rhodium, was significantly improved under polarization by constant current. The stability was increased by one order of magnitude when pure ruthenium coating was replaced by $Ru_{0.7}Rh_{0.3}$. The stability was further improved, for an additional order of magnitude (from 20 to 240 min), using $Ru_{0.3}Rh_{0.7}$ coating (Table III). The most stable was $Ru_{0.2}Rh_{0.8}$ coating, which after six hours of galvanostatic polarization by 0.12 A cm^{-2} current density did not exhibit the sharp change of potential, i.e., a complete dissolution. As seen in Fig. 6, the electroactive surface area was decreased by about 30%.

It should be pointed out that stability tests carried out using RRDE (Fig. 4), and those presented in Fig. 5, cannot be directly compared. The dissolution of electrodeposited ruthenium on platinum was faster under potential cycling conditions using also RRDE measurements [14], where we have shown that ring current at 1.2V dropped significantly while potential was held at 1.2 V. In other words, the dissolution was faster under potential cycling conditions (Fig. 4) than under constant potential which was established during galvanostatic polarization in the experiments presented in Fig. 5.

5. Conclusions

Mixed Ru-Rh coatings can be produced by electrodeposition on titanium. Due to higher current efficiency of rhodium electroplating, the electroactive real surface area was increased by the increase of the rhodium content in the coating.

The high electrocatalytic activity of electrodeposited ruthenium coating on titanium in the oxygen evolution reaction from an acid solution is accompanied by dissolution of the ruthenium oxide layer at the potential more positive than 1.2 V vs. SCE.

The addition of rhodium and formation of mixed Ru-Rh coating stabilized the surface layer and increased the service-life of $Ru_{0.2}Rh_{0.8}$ coating 800 times compared to pure ruthenium coating.

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