

An approach to the effect of hydrogen loading on the anodic behaviour of Ti in NaOH solutions

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The voltammetric behaviour of titanium electrode in 0.5 M NaOH solution was investigated. The I-E curves revealed the presence of a well-defined O₂ evolution peak. The peak current density decreases to a great extent by pre-loading the metal with hydrogen by cathodic polarization, indicating that pre-loading the metal with hydrogen suppresses the O₂ evolution. The oxidation of hydrogen loaded inside the metal was suggested to be responsible for reducing the O₂ evolution peak current density during Ti anodization.

The charge consumed during the anodic polarization of Ti, below O₂ evolution potential, was used to estimate the anodization coefficient and capacitance measurement was conducted for measuring the dielectric constant of the oxide film. Pre-loading the metal with hydrogen was found to decrease the oxide film formation rate as well as the dielectric constant.

1 Introduction

The growth of the anodic oxide films on titanium was extensively studied [1–8]. Most of the studies concentrated on the anodization of titanium in acidic media. On the other hand, some work was carried out on the behaviour of titanium in alkaline media [3–5]. In the anodic region, the final product of the electrode oxidation is mainly TiO₂ [2, 6, 7]. O₂ evolution was always observed during titanium anodization. *Dyers* and *Leach* [1] attribute O₂ evolution to the increase in electronic conductivity of the film that accompanies the crystallographic transformation. It was recently suggested that O₂ evolution cause a variation in the dielectric constant of titanium oxide [8].

Ti is known to absorb hydrogen. During cathodic polarization, in the potential range of hydrogen evolution, the formation of titanium hydride is an established phenomenon [9–12]. Several techniques have been used to study the composition and morphology of the titanium hydride phases [12, 13]. The presence of an oxide layer on the metal surface during cathodic polarization results in: a) film dissolution [11, 14], and b) electrochemical film loading with hydrogen species [4, 14–16]. Three modes of electrochemical incorporation of hydrogen in the film were proposed [17]: i) a small amount reversibly absorbed that can be removed from the film by slight changes in potential, ii) a large amount that can be removed only under illumination, and iii) an unknown amount that becomes bound to the lattice and cannot be removed with anodic photobleaching. Different authors [17–19] have reported the loading of the TiO₂ films with hydrogen indicating an increase in the film conductivity [15]. Due to this process, and a large change in the dielectric constant was observed [17, 20]. In addition, it was suggested that some species of hydrogen remain in the oxide phase modifying its physicochemical properties [16].

In a previous paper [5], the pH effect on the anodic behaviour of titanium was studied. A comparison was made using the thermodynamic data that revealed the possibility of formation of TiO₂ directly from titanium hydride. The purpose of the first part of this paper is to investigate the anodic behaviour of titanium in NaOH solution. The second part presents

the influence of hydrogen loading into the metal on the O₂ evolution reaction, on the growth rate of oxide and finally on the dielectric constant of the oxide film.

2 Experimental

Experiments were carried out in a conventional glass cell that was described elsewhere [5]. The titanium (Koch light 99.9%) working electrodes used were prepared in the same manner as in a previous work [8]. The auxiliary electrode was a Pt wire separated from the electrolyte solution by sintered glass. The reference electrode was Hg/HgO (1.0 M NaOH) to which, unless otherwise stated, all potential values are referred. The reference electrode was placed into a glass jacket provided with a Luggen capillary. 0.5 M NaOH solutions was prepared from a concentrated stock solution by appropriate dilution using a triply distilled water. The experimental results were obtained by employing the cyclic voltammetric technique. Mechanically polished titanium electrodes were polarized in NaOH solutions at different scan rates. At each scan rate, the potential sweep started at –0.8 V in the positive direction and ended at +5 V, then scanned back to –0.8 V in the cathodic direction.

A set of cyclic voltammetric I-E curves were performed on Ti in 0.5 M NaOH solutions at a single scan rate of 10 mV s⁻¹. In these experiments, the potential was scanned from –0.8 to 0.2 V in the anodic direction and then reversed to 0.0 V. At this potential, the polarization was switched off and the open circuit capacitance of the formed oxide was immediately measured at a fixed frequency of 1.0 kHz (a conventional a.c. bridge was used [8]). The procedure was repeated reaching a different higher value of potential each time in the anodic direction, before reversing the scan and measuring the capacitance at 0.0 V. A similar set of experiments was also carried out, in which the electrode was cathodically polarized at a potential value of –1.8 V for 30 min before starting the anodization process at –0.8 V as in the previous set. In both sets, values of the anodic charge were simultaneously measured at each formation potential.

The electrochemical measurements were performed using system AMEL 5000 (supplied by Amel instrument, ITALY). A PC was interfaced with the instrument through a serial RS-232C card. Amel Easyscan software was used in connection with the PC to control the Amel 5000 system. All the reported potentials were corrected up to 90% of its value by the positive feedback technique.

3 Results and Discussion

3.1 Behaviour of titanium electrode in NaOH solution

Cyclic voltammetric I-E curves of Fig. (1) show that Ti anodization in the NaOH solutions are basically different from those found in other electrolytes studied before (acidic sulphate and buffer solution [5]). Two major differences are observed in connection with the voltammograms in NaOH compared to other electrolytes:

- At one and the same scan rate the current densities of the passivation peaks (the first plateau in Fig. (1) in case of NaOH are much higher than those in other electrolytes.
- A well-defined anodic peak appears in the voltammograms in NaOH at about 1.5 V vs NHE. In other electrolytes, the corresponding peak appears as a small kink at about 1.8 V vs NHE. This peak was interpreted as a partial O₂ evolution due to a temporary breakdown of the oxide surface layer. The proper O₂ evolution starts at about 3 V vs NHE characterized by a sharp increase of current density extending up to 5 V before reversing the scan.

The low current densities accompanying the passivation in the voltammogram of titanium in acidic and neutral electro-

lytes is possibly due to one of the following or both: (i) higher rate of oxide film dissolution in acidic-neutral electrolytes [21] and (ii) incorporation of anions other than OH⁻ into the oxide. Anion incorporation was found to decrease the oxide film formation rate [22].

The following regions can be recognized in Fig. (1):

1. A first anodic plateau, (I).
2. A well-defined anodic peak, probably due to O₂ evolution, (II).
3. A second anodic plateau of a higher current density value than that of the first one, (III).
4. Nearly zero current density values in the cathodic direction after reversing the scan, (IV).

The first anodic plateau (I) is connected to the anodic oxidation of the titanium metal to TiO₂ [2, 6, 7]. With increasing potential, a transition from amorphous to crystalline state of the passive film takes place [23]. Due to this morphological transformation, small flaws in the oxide may form, through which the electronic conductivity of the film increases and O₂ gas evolves. Similar assumption was reported by Dyer *et al.* [1]. O₂ bubbles were always observed on the electrode surface at a potential value of about 1.0 volt at all scan rates studied. From the curves of Fig. (1), the O₂ evolution peak (II) potential was found to shift to more positive values with the increase of scan rate. Scanning the anodization potential to more positive values, the oxide flaws are repaired and oxide thickening continues leading finally to the second anodic plateau (III). The rate of oxide film formation becomes higher at the potential region above O₂ evolution. This is due to the catalytic effect of oxygen gases on the rate of oxide growth [8]. This could explain the higher value of the current density of the second plateau than that of the first one [see Fig. (1)]. With respect to Ti anodization in acidic and neutral electrolytes, the proper O₂ evolution takes place at about 3 V vs NHE. At this potential O₂ evolution and the oxide film thickening take place simultaneously.

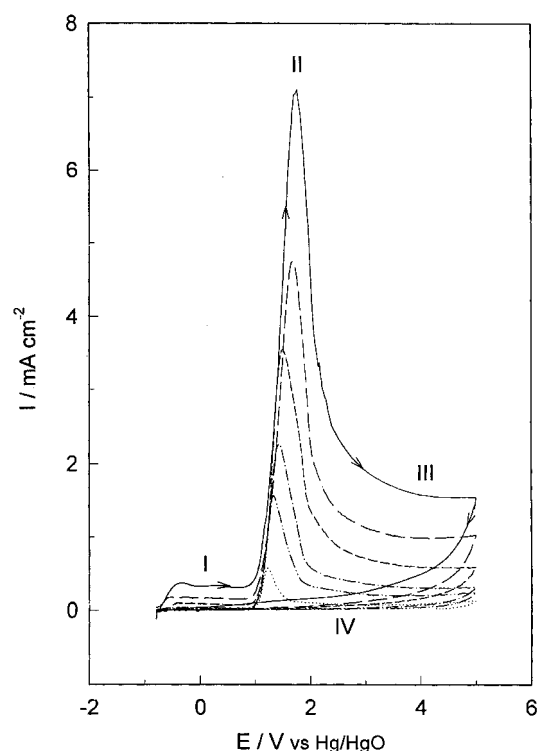


Fig. 1. Cyclic voltammograms of mechanically polished Ti electrodes in 0.5 M NaOH solutions at scan rates (in mV s⁻¹): — 100, — — 50, - - - 25, — — 10, — — 5, and1.

3.2 Effect of hydrogen loading

Hydrogen loading of titanium in NaOH solutions was achieved by cathodic polarization in the range of potential corresponding to the hydrogen evolution reaction (negative than -0.8 V vs Hg/HgO). Fig. (2) presents the voltammetric curves at 50 mV s⁻¹ recorded first in the potential range of -0.4 to 5.0 V vs Hg/HgO and then by moving the starting potential by 200 mV steps down to -1.8 V. It is interesting in this figure to note that the starting potential affects the anodic peak current density of O₂ evolution. Varying the starting potential from -0.4 to -1.0 V causes an increase in the peak current density of O₂ evolution, Fig. (2a). After exceeding -1.0 V, as a starting potential, the peak current density of O₂ evolution starts to decrease (I in Fig. (2b)). At highly negative starting potentials a broad peak results (II in Fig. (2b)), its potential is shifted to a more positive value than that of peak I.

On the basis of the results of Fig. (2), the increase of starting potential in the cathodic direction from -0.4 to -1.0 V results in increasing the polarization time in the passive region. So, it is expected to have a higher oxide thickness with increasing the potential window of polarization. It was reported above that O₂ gas evolves due to the presence of flaws in the oxide film formed as a result of its morphological transformation. The repair of these flaws could proceed faster for thin oxides. Accordingly, for thicker oxides the slower rate

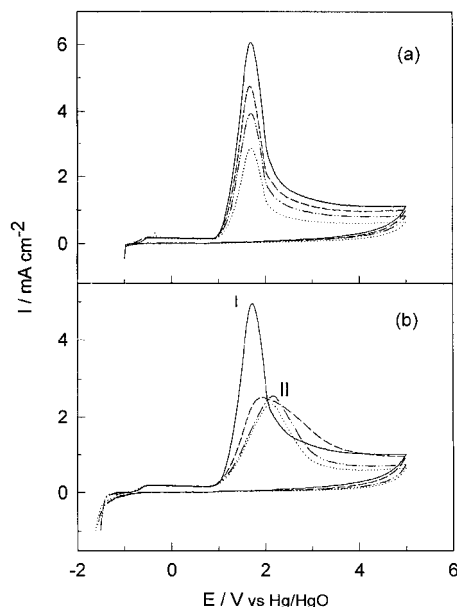


Fig. 2. Cyclic voltammograms of Ti electrodes at a scan rate of 50 mV s^{-1} with different starting potentials: (a) -0.4 V , — — — -0.6 V , - - - -0.8 V and — — — -1.0 V vs Hg/HgO. (b) — — — -1.2 V , - - - -1.4 V , — — — -1.6 V and -1.8 V vs Hg/HgO.

of the repair process could lead to higher O_2 evolution currents. This can explain the increase of the peak current density of O_2 evolution upon varying the starting potential from -0.4 to -1.0 V [see Fig. (2a)]. The effect of hydrogen loading appears when exceeding -1.0 V of the starting potential in the cathodic direction (beginning of H_2 evolution). The O_2 evolution peak current density decreases, becomes broad and shifts to more positive values especially in the range of potential from -1.4 to -1.8 V . In this potential range the results show another anodic peak merged with the O_2 evolution peak.

Under cathodic polarization of titanium in the hydrogen evolution potential region, hydrogen can be electrochemically incorporated into the metal either in the form of atomic hydrogen or becomes bound to the metal in the form of hydride. The results of Fig. (2b) show that hydrogen loading causes the suppression of O_2 evolution. The more the amount of loaded hydrogen, the more evident is this process. In order to explain this phenomenon the following explanations can be relevant:

1. Oxidation of the loaded atomic hydrogen in the anodic scanning may consume part of the anodic charge and thus represents a parallel reaction to the O_2 evolution.
2. O_2 evolution is an electrochemical process that proceeds initially by the adsorption of O^{2-} on the electrode surface. Part of the loaded hydrogen can diffuse into the oxide lattice and dissociates into electrons and protons [24]. These protons are then expelled by the applied anodic potentials to reach the oxide surface where they can interact with part of the adsorbed O^{2-} forming H_2O .

The influence of the amount of loaded hydrogen on the O_2 evolution process is demonstrated in Fig. (3). Potential holding at different time intervals in the hydrogen evolution region was carried out before sweeping in the positive direction. The results of Fig. (3a) show that, even five minutes holding at the hydrogen evolution potential leads to a large decrease in the current density values of the O_2 evolution peak (about 70%

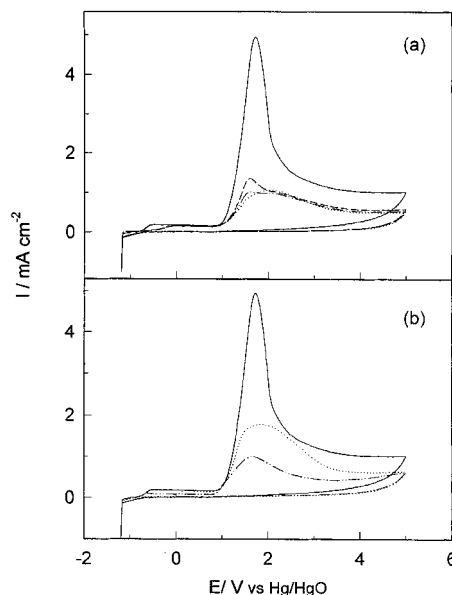


Fig. 3. Cyclic voltammograms of Ti electrodes at a scan rate of 50 mV s^{-1} . (a) Potential sweep started at -1.2 V after: — 0 min., - - - 5 min., — — — 10 min. and 30 min. time holding at -1.2 V ; (b) — Potential sweep started at -0.8 V after 30 min. time holding at -1.8 V Potential sweep started at -0.8 V after 30 min. time holding at -1.8 V and then left stand in the electrolyte for 3 h.

lower). The voltammetric curve at a 5 minutes time holding shows a second anodic peak. This peak might be interpreted as the oxidation of incorporated hydrogen that partly remains inside the metal. A much longer holding time in the hydrogen evolution potential region [see Fig. (4a)] leads finally to a broad peak. This peak might possibly correspond to two anodic peaks merged together, one for the O_2 evolution process and the other for the hydrogen oxidation.

It is also noted in Fig. (3) that the voltammograms performed with time holding at the hydrogen evolution potential region have relatively lower passivation current density values (the first anodic plateau) than that of the voltammograms performed with no time holding. Furthermore, the passivation process starts at more positive potential values upon loading the metal with hydrogen. This effect is clearly shown in Fig. (3b). This figure represents a comparison between an experiment carried out on titanium in 0.5 M NaOH at a starting potential of -1.2 V without time holding and another at the same starting potential but with a preceding time holding at -1.8 V for 30 min. Suppression of O_2 evolution to about 80% was detected, upon hydrogen loading, as shown from the difference between the peak current density values for O_2 evolution of the two curves. Also, the current density value of the first plateau is about 50% lower in the hydrogen-loaded curve than in the unloaded one. This leads to the conclusion that early loading of the metal with hydrogen may alter the oxide film formation rate (see below). If the preloaded electrode is just left standing in the electrolyte the loading effect of hydrogen starts to disappear in a few hours, see Fig. (3b).

3.2.1 Capacitance measurements

The following relation can relate the measured capacitance to the oxide formation potential [25]:

$$\frac{1}{C} = \frac{\alpha}{\epsilon \epsilon_0 r} (E_f - E_{ox}) \quad (1)$$

where C is the measured capacitance, α is the anodization coefficient, ϵ is the dielectric constant of the oxide, $\epsilon_0 = 8.85 \times 10^{-14} \text{ F cm}^{-1}$ is the dielectric constant of vacuum [26], r is the roughness factor, E_f is the oxide formation potential and E_{ox} is the potential of the beginning of oxide formation. Fig. (4) presents the relationship between the reciprocal capacitance, $1/C$, and the formation potential, E_f , for oxides formed on loaded Ti electrodes as well as for oxides formed on the unloaded electrodes. The figure shows the following:

1. For one and the same formation potential, the values of capacitance of oxides formed on the loaded Ti electrodes are always higher than the capacitance of oxides formed on the unloaded electrodes.
2. One straight line of a lower slope in the potential range below O_2 evolution potential (extending upto 0.8 V, region I) and another straight line of a higher slope obtained at higher potentials (extending from 1.0 to 3.5 V, region II).
3. The slopes of the straight lines of the loaded set of experiments are always lower than of the unloaded set.

It is suggested that the different slopes obtained in Fig. (4) can be due to changes either in the value of the anodization coefficient α , in the value of the dielectric constant ϵ of the oxide film or both simultaneously.

3.2.2 Estimation of the anodization coefficient

The graphical presentation of the relation between the anodic charge, Q , and formation potential, E_f , is given in Fig. (5) for both of the unloaded and loaded electrodes. It is noted from this figure that at one and the same formation potential the value of the anodic charge in case of the loaded

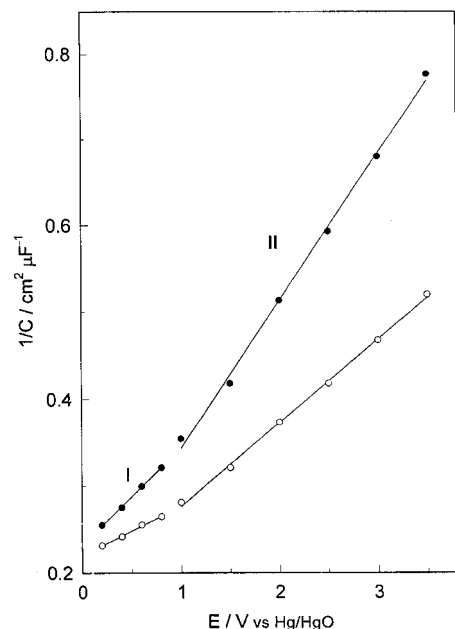


Fig. 4. Relation between the reciprocal capacitance and the formation potential of Ti anodization without hydrogen loading ● and with hydrogen loading ○ at -1.8 V for 30 min.

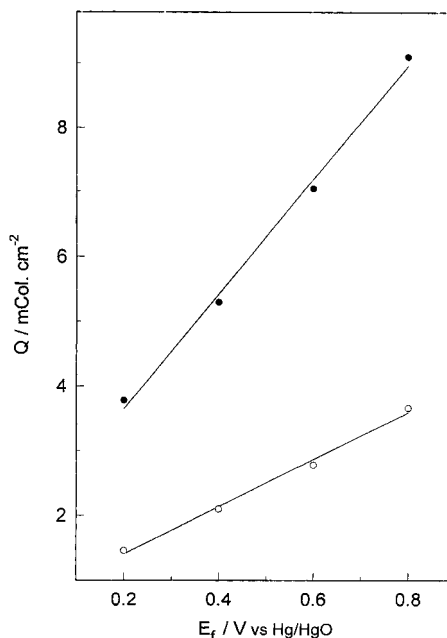


Fig. 5. Relation between the anodic charge and formation potential of Ti anodization without hydrogen loading ● and with hydrogen loading ○ at -1.8 V for 30 min.

set is always lower than that in the other set. From the slopes of the obtained straight lines one can calculate the anodization coefficient, α (in nm V^{-1}), of the oxide growth according to the following equation [27]:

$$\alpha = \frac{d Q_{ox}}{d E_f} \frac{M}{nF \delta_{ox}} \quad (2)$$

where Q is the anodic charge density, E_f is the formation potential, M is the molecular weight of the oxide, δ_{ox} is the film density, n is the number of electrons exchanged and $F = 96499 \text{ C mol}^{-1}$. The quantity $M/nF \delta_{ox}$ is the volume of oxide formed per coulomb and has, in this case, a value of $5.37 \times 10^{-5} \text{ cm}^3 \text{ C}^{-1}$ [28]. The value of α obtained for both sets of experiments was calculated only in the potential range below O_2 evolution potential ($E_f < 1.0 \text{ V}$) assuming 100% current efficiency and also assuming that δ_{ox} remains constant. Values of α are 4.76 and 1.96 nm V^{-1} for the oxides formed on the hydrogen unloaded and loaded Ti electrodes, respectively. The values of α thus obtained confirm that loading the metal with hydrogen causes a structural changes in the oxide film. A structural changes in the oxide film apparently produced different slope of the $1/C$ vs E_f plot, see Fig. (4). When Ti is polarized cathodically in the hydrogen evolution potential range, a hydride layer is formed [9]. The lower values of α obtained for the oxide formed on the loaded Ti electrodes could be due to:

- An error due to the estimation of 100% current efficiency for this type of oxide.
- Different oxide density δ_{ox} .
- Oxide formed on Ti in the range of hydrogen evolution potential is possibly a mixture of oxide and hydride. At more positive potential values TiO_2 can be directly formed from titanium hydride in acidic and neutral solutions (for hydrogen loaded Ti electrodes) [8].

3.2.3 Estimation of the oxide film thickness and its dielectric constant

The oxide film thickness formed in the potential range below O₂ evolution potential can be estimated using the following relation [27]:

$$d = d_0 + \frac{M}{nF \delta_{ox}} Q_{ox} \quad (3)$$

where d is the oxide film thickness and d_0 is the initial film thickness (a value of 1.0 nm was used [29]). A plot of the film thickness against $1/C$ is shown in Fig. (6). From the slope of the straight lines obtained, the dielectric constant values are calculated using the relation:

$$d = \frac{\epsilon a}{(4 \pi) 9 \times 10^9} \frac{1}{C} \quad (4)$$

where C (Farad) is the capacitance, ϵ is the dielectric constant and a (m²) is the electrode area. Values of 49.0 and 38.7 were obtained for the dielectric constant of oxides formed on the hydrogen unloaded and loaded Ti electrodes, respectively. The lower value of the dielectric constant of the oxide formed on the hydrogenloaded metal is an indication of its higher conductivity. The conductivity induced by generation of hydrogen is attributed to the diffusion of hydrogen atoms into the crystal where they donate their electrons to the conduction band, leaving interstitial proton [18]. A large change in the dielectric constant was also illustrated previously due to the loading of the TiO₂ films with hydrogen [17, 20]. It was established earlier [25] that a relatively lower dielectric constant, and consequently, a higher conductivity might accompany lower rates of oxide film formation. Using the obtained values of the dielectric constant, the oxide growth rate can also be estimated from equation (1). Table (1) represents the different values of α calculated using equations (1) and (2)

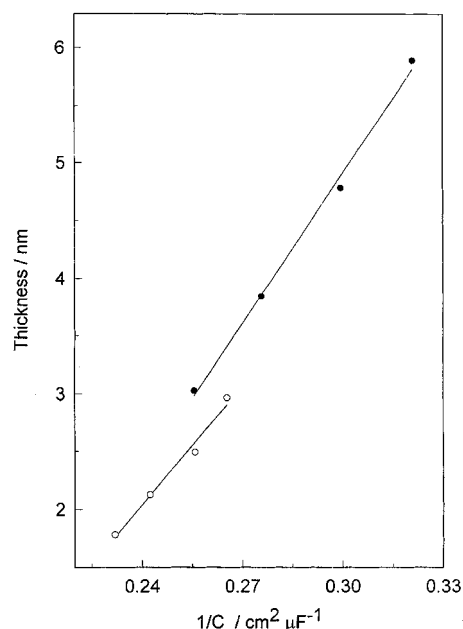


Fig. 6. Relation between the oxide thickness and the reciprocal of capacitance of Ti anodization without hydrogen loading ● and with hydrogen loading ○ at -1.8 V for 30 min.

and also values of the dielectric constant of TiO₂. It is noted that the obtained values of α/r using equation (2) are in excellent agreement with those obtained from Fig. (5) equation (1).

4 Conclusions

The experimental studies in this work suggest that the behaviour of Ti in NaOH solutions differ from that in acidic and neutral electrolytes in some points. The rate of oxide film formation in NaOH is higher as well as the O₂ evolution reaction. Loading the metal with hydrogen leads to suppressing the O₂ evolution process. This phenomenon was interpreted as due to a competitive oxidation of the loaded hydrogen during anodization.

Loading the metal with hydrogen results also in altering the oxide film formation rate. It is concluded that this effect could be due to the hydrogen oxidation (hydrogen still loaded) that consumes part of the electric charge. In addition, a lower dielectric constant value was computed for oxides formed on Ti metal preloaded with hydrogen. Hydrogen loading in the metal could possibly increase the conductivity of the electrochemically formed oxide by increasing the donor impurities and donating electrons to the conduction band.

Table 1. Estimated values of the anodization coefficient α and the dielectric constant ϵ for Ti oxides formed anodically in 0.5 M NaOH in the range of potential below O₂ evolution.

	Oxide formed on the hydrogen unloaded metal	Oxide formed on the hydrogen loaded metal
ϵ	49.0	38.7
α^\dagger	4.76	1.96
α/r^*	4.74	1.95

† Calculated using equation (2)

* Calculated using equation (1)

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Received: 13/11/98

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DVS-Veranstaltungen

Roboter '99 - Eine erfolgreiche DVS-Tagung

zur Automatisierung von Schweiß- und Fügeprozessen

Die 5. DVS-Sondertagung „Roboter'99“ mit dem Titel „Neue Wege zum wirtschaftlichen Schweißen mit Robotern und Automaten“ bot am 9. und 10. Juni 1999 in Fellbach für interessierte Fachleute ein umfassendes Programm zum Stand der Automatisierung beim Schweißen sowie bei kombinierten Fügeprozessen.

Nach einem Überblick über den Stand und die Tendenzen der Roboteranwendung wurde die methodische Einsatzplanung durch Roboterhersteller und Serviceunternehmen vorgestellt, die durch mehrere Beispiele von Anwendungen in kleinen Unternehmen belegt werden konnte. Neben der Vorstellung neuer Mechanisierungsgeräte und Steuerungsmaßnahmen zur Qualitätssicherung wurde ausführlich der produktive Einsatz fortgeschrittener Anwendungen in den Bereichen „Fügen im Leichtbau“ sowie „Schweißen mit Hochleistungslichtbogen- und Strahlverfahren“ dargestellt. Besonderen Anklang fand die Themengruppe „Programmieren, Bedienen und Qualifizieren“, in der sowohl Verbesserungen der Roboterprogrammierung als auch die Integration und Qualifizierung der Mitarbeiter ausführlich diskutiert wurden.

Das Vortragsprogramm der Tagung ist im DVS-Bericht Band 199 wiedergegeben.

Auf der in die Tagung integrierten Firmenleistungsschau, die insbesondere von Praktikern aus der Region gut besucht war, führten bedeutende Hersteller von Robotersystemen und Peripheriegeräten den Einsatz moderner Anlagen und -komponenten in der Praxis vor.

Parallel zur nächsten DVS-Sondertagung „Roboter“, die am 12. und 13. Juni 2002 stattfinden wird, ist daher wieder eine umfangreiche Firmenleistungsschau geplant.

Ihre Ansprechpartnerin beim DVS:
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Große Schweißtechnische Tagung in Weimar

Als größte nationale Fachtagung auf dem Gebiet der Schweißtechnik findet vom 15. bis 17. September 1999 die 52. Große Schweißtechnische Tagung des DVS - Deutscher Verband für Schweißen und verwandte Verfahren e.V. in Weimar statt.

Schweißtechnische Toptechnologien werden in anwendungsbezogenen aktuellen Vorträgen wie „Gütesicherung von Schweißarbeiten beim Neubau von Schienenfahrzeugen“ oder in Vortragsblöcken wie „Sanierung von Anlagen und Bauwerken mit Hilfe schweißtechnischer Verfahren“ von Fachleuten namhafter Unternehmen und Institute präsentiert.

Als besonderes Highlight hält Herr Dr. Lothar Späth, Vorstandsvorsitzender der JENOPTIK AG, einen Festvortrag zum Thema „Die deutsche Wirtschaft im europäischen und globalen Umfeld“.

Im Rahmen der Großen Schweißtechnischen Tagung findet auch die Abschlusspräsentation des vom Bundesministerium für Bildung und Forschung geförderten Projektverbundes „Qualifizierung von Laserverfahren Laser 2000“ statt.

Das Tagungsprogramm sowie weitere Informationen können unter der nachfolgend aufgeführten Anschrift abgerufen werden.

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