COMPOSITION AND THERMODYNAMIC CHARACTERISTICS OF HIGHER-VALENCE NICKEL OXIDES

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It is shown that the handbook values of the free energy associated with the formation of higher-valence nickel oxides, as given by V. Latimer and M. Pourbaix, have been determined incorrectly on the basis of a wrong interpretation of the reactions occurring at the nickel-oxide electrode of an alkaline storage battery. The free energy of β-NiOOH (−77.5 kcal/mole) is much more positive and there is no evidence that higher-valence oxides exist. Prevailing concepts about the role of higher-valence oxides in the passivation of nickel ought to be revised.

The limits of electrode processes as well as their mechanisms (passivation and activation of nickel, evolution and ionization of oxygen, etc.) in which water participates or is formed on either the nickel or the nickel-oxide electrode cannot be precisely established without a knowledge of the thermodynamic characteristics of nickel oxides and nickel hydroxide. In the final analysis, modern concepts about these processes depend implicitly on arbitrarily interpreted measurements made on the nickel-oxide electrode of an alkaline battery and, therefore, it is precisely on the basis of such measurements [1-5], supplemented with series of assumptions, that the characteristics of nickel-oxide compounds have been tabulated in special thermodynamic charts [6, 7], in general thermodynamic charts [8], in handbooks, and in separate reports [9, 10]. The object of this study is to reveal hitherto hidden inaccuracies in tabulated values and indicate the feasibility of some refinement.

Although the various authors use almost identical test data as the basis for their thermodynamic studies, they greatly differ not only in the evaluation of the equilibrium potential associated with the formation of phase oxides of nickel but also in the very determination of the stoichiometric series of such compounds (Table 1). This suggests that each of the proposed series is a hypothetical one. One of them includes four stoichiometric oxides: $NiO-Ni_3O_4-Ni_2O_3-NiO_2$, with the middle transition $Ni_3O_4 \rightleftharpoons Ni_2O_3$ regarded as the principal current-generating process at the nickel-oxide electrode. The other series [7, 9] consists of only the two extreme members of this one and the transition between them is, accordingly, regarded as the current-generating process.

In order to determine to what extent these hypotheses correspond to reality, we consider a diagram (Fig. 1) where the ordinates of horizontal segments (renumbered in accordance with the reactions listed in Table 1) represent the equilibrium potentials of the said phase transitions in 1.0 N alkali and their end points indicate the relative amounts of reduced and oxidized phases, respectively, in terms of the atomic ratio O:Ni in the anhydrous oxide (i.e., in terms of the index n in the tentative formula NiO_n).

Several attempts have been made to obtain a potential-composition equilibrium diagram for a nickel-oxide electrode [11-15]. In [11], e.g., this was done for several compositions of a nickel-oxide electrode by plotting pairs of curves of spontaneous potential changes, after anodic and cathodic polarization had been respectively discontinued, and then extrapolating both curves in $E-\log\tau$ coordinates to their intersection at the point regarded as corresponding to the equilibrium potential for the given composition. This has resulted in curve E (Fig. 1). In [12], on the other hand, as the equilibrium composition for a given potential was regarded the composition of the nickel-oxide film electrode resulting from its potentiostatic treatment for a period necessary to reach steady state. This has yielded curve P (Fig. 1). The authors of this study measured the steady-state potentials and determined the composition of the active mass of nickel-oxide electrodes with a metal–ceramic structure, after they had been held for the period of one month without current. Our procedure has yielded curve V (Fig. 1).

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TABLE 1. Equilibrium Reactions and Equations Characterizing the Nickel Oxides–Water System

No.	Reactions	Equilibrium potentials, V	Equilib. potent. at pH = 14, V	Literature references
1	$Ni_3O_4+2H^++2e=3NiO+H_2O$	0,876–0,059 pH	0,048	[6]
2	$Ni_2O_3+2H^++2e=2NiO+H_2O$	1,020–0,059 pH	0,192	[6]
3	$Ni_2O+2H^++2e=NiO+H_2O$	1,226–0,059 pH	0,398	[6]
4	$3Ni_2O_3+2H^++2e=2Ni_3O_4+H_2O$	1,305–0,059 pH	0,477	[6]
5	$NiO_2+4H^++4e=Ni_3O_4+2H_2O$	1,401–0,059 pH	0,573	[6]
6	$2NiO_2+2H^++2e=Ni_2O_3+H_2O$	1,434–0,059 pH	0,608	[6]
7	$NiO_2+2H^++2e=Ni(OH)_2$	1,588–0,059 pH	0,760	[9]
8	$NiO_2+2H^++2e=Ni(OH)_2$	1,318–0,059 pH	0,490	[7]
9	$Ni_2O_3+6H^++2e=2Ni^{2+}+3H_2O$	1,753–0,177 pH –0,0591 lg $a_{Ni^{2+}}$		[6]
10	$NiO_2+4H^++2e=Ni^{2+}+2H_2O$	1,593–0,1182 pH –0,0295 lg $a_{Ni^{2+}}$		[6]
11	$NiO_2+4H^++2e=Ni^{2+}+2H_2O$	1,93–2,04– –0,1182 pH – –0,0295 lg $a_{Ni^{2+}}$		[9, 10]
12	$NiOOH+H^++e=Ni(OH)_2$	1,333–0,059 pH	0,505	This report
13	$NiOOH+3H^++e=Ni^{2+}+2H_2O$	2,04–0,177 pH – –0,059 lg $a_{Ni^{2+}}$		The same

The set of these three curves, obtained at different times by different authors according to different methods with different electrode specimens, clearly indicates that the degree of oxidation of a nickel-oxide electrode within the $1.10 < n < 1.45$ range has almost no effect on the equilibrium potential. At least, the relation between them is so smooth, monotonic, and weak as to not reveal any difference between the current-generating electrode reaction and the classical phase transition which, moreover, remains the only one throughout the interval. The equilibrium potential of this transition is $E = 0.505 \pm 0.02$ V.

If a single phase transition covers the range of n from 1.45 to 1.10, however, then it follows from the condition of material balance at any degree of transformation $\leq 100\%$ between two phases that the lower-valence one cannot have $n > 1.10$ and the higher-valence one cannot have $n < 1.45$. Among all compounds in the stoichiometric series, only those with $n = 1$ (NiO and $Ni(OH)_2$) satisfy the first requirement and only those with $n = 1.5$ (Ni_2O_3 and NiOOH) or $n = 2$ (NiO_2) satisfy the second requirement. Regardless of the calculated values of the potential, therefore, the material balance identifies the state of nickel in the reduced phase as divalent and excludes the possibility of a phase with Ni_3O_4 ($n = 1.33$) appearing as an initial or final product of the current-generating phase transition.

It is appropriate to point out here that the existence of a so-called second discharge stage, noted by several authors [1, 2, 6, 16], during deep discharge in the form of a longer or shorter potential delay within the range from +0.05 to −0.10 V on the discharge curve for a nickel-oxide electrode, had for a long time been regarded as the main proof of Ni_3O_4 participating in the action of a nickel-oxide electrode. Eventually, however, such an interpretation of this delay was found wrong. The potential at which such a delay occurs depends largely on the material of the current feeder, while on platinum such a delay appears [16] also in the absence of nickel oxides. The cathodic process occurring at the potential of this second delay directly at the current feeder was subsequently [17] shown to be reduction of oxygen dissolved in the alkali and formation of hydrogen peroxide. The lengthy duration of this process is explained by the fact that a part of the nickel oxides undergoing reduction in the main discharge stage has not yet been reduced while it loses contact with the current conductor. These oxides immediately enter into a chemical reaction with the hydrogen peroxide, become reduced to $Ni(OH)_2$ and give off the same amount of oxygen which was reduced to peroxide in the preceding stage of the process. In this way, the same nickel oxides are reduced and formed in the second discharge stage as in the first, but here the loss of direct contact with the current feeder makes the reduction in the presence of dissolved oxygen and hydrogen peroxide proceed irreversibly.

This conclusion has been fully confirmed by numerous structural x-ray examinations [13] revealing that $Ni(OH)_2$ is the principal phase in a discharged nickel-oxide electrode and hardly exists in a charged one, but is certain to form in the latter already during mild cathodic polarization which does not shift the electrode potential below 0.48 V. On the contrary, only once [2] and then only on an x-ray photograph of a long-stored nickel-oxide electrode were there found lines attributed to Ni_3O_4 hydrate; this finding has never been confirmed again throughout the last 25 years [18-20]. A NiO_2 phase has also not been revealed by x-ray photographs of a nickel-oxide

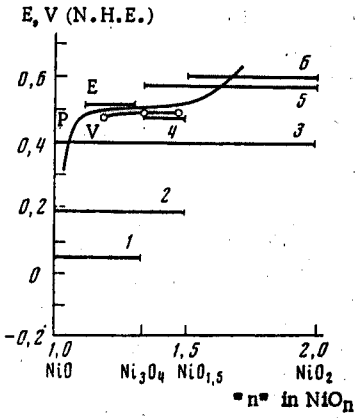

Fig. 1. Equilibrium potential of the nickel-oxide electrode as a function of the relative amount of higher-valence nickel oxides in it: 1-6) segments correspond respectively to reactions 1-6 listed in Table 1, E) based on data in [11], P) based on data in [12], V) steady-state potentials of the nickel-oxide electrode.

electrode regardless of the charge level,* while the product of $Ni(OH)_2$ oxidation has been identified as β-NiOOH, i.e., the principal component of a charged nickel-oxide electrode [2,18-20].

All this identifies the basic potential-establishing process at a nickel-oxide electrode as an electrochemical phase transition

$$Ni(OH)_2+OH^- \rightleftharpoons \beta\text{-}NiOOH+H_2O+e, \qquad (12)$$

which, according to classical analyses of concentration changes near an electrode [21] and according to direct radioisotopic measurements [22], amounts to an anodic extraction of hydrogen from the lower-valence nickel hydroxide and its reverse cathodic infusion into the higher-valence nickel hydroxide.

An alternative attempt to attribute the primary potential of a nickel-oxide electrode not to reaction (12) but to a direct transition between stoichiometric oxides of divalent and tetravalent nickel must be rejected. Such a transition bypassing the intermediate-valence phase would be theoretically probable, but only if the trivalent oxide did not form 1) either on account of its thermodynamic instability (even when forming then, it would have to spontaneously dissociate into the higher-valence oxide and the lower-valence one) or 2) on account of peculiar kinetic (steric) obstacles which prevent its seeding and buildup as an independent phase. In this case the active mass of a nickel-oxide electrode charged to n = 1.5 would have to consist of divalent and tetravalent nickel oxides in approximately equal amounts, with almost no NiOOH present. This would, in accordance with what has been said earlier, contradict all experimental evidence.

One must also reject the hypothesis that some amount of NiO_2, though insignificantly small and not perceptible by x-radiographic examination, can form along with β-NiOOH within the $1.0 < n < 1.5$ range. This would have been possible only if the equilibrium potentials of $Ni(OH)_2$ oxidation to β-NiOOH and to NiO_2 respectively were close. According to Luther's rule, however, at approximately the same potential there should then also a β-NiOOH $\rightleftharpoons$ NiO_2 transition necessarily corresponding to a flat segment along the charge–discharge curve within the $n > 1.5$ range. In reality a further increase of n beyond n = 1.45 is accompanied by a fast monotonic increase of the derivative dE/dn and, therefore, oxidation altogether does not continue beyond n = 1.6, at which point the anode current begins to be almost completely expended on the evolution of O_2.

In view of what has been said earlier, this clearly indicates that, while some amount of nickel with a valence higher than three exists in a highly charged nickel-oxide electrode, it does not appear in the form of a stoichiometric phase compound but as a solid solution in the β-NiOOH matrix, and here its activity increases with its concentration.

*One author [9] tried to prove the existence of NiO_2 by erroneously referring to the report [5], where in fact only some unidentified and unanalyzed black nickel oxide had been found on platinum at 0.76 V. The same oxides form much easier on a nickel or peroxide–lead anode and their electrode potential in the zero-current state is 0.48 V, very close to the primary potential of a nickel-oxide electrode.

Does, somewhere within the range of intensive oxygen evolution, this solid solution reach its homogeneity limit beyond which an independent NiO_2 phase precipitates? The unsuccessful attempts to reveal such a phase by x-ray examination can be reconciled with this hypothesis, if one assumes that this phase immediately after formation decomposes with an evolution of oxygen. A nonstoichiometric solid solution can also decompose, by the way, but checking such hypotheses experimentally is not feasible at this time. It may be stated firmly, however, that (Fig. 1) the equilibrium potential of β-NiOOH oxidation to NiO_2 in 1.0 N alkali is under no circumstances lower than 0.7 V and, therefore, the stoichiometric oxide of tetravalent nickel does not at all participate in the action of a nickel-oxide electrode and certainly not its hypothetical hydrate $Ni(OH)_4$ [10], which in principle cannot be formed by even the kinetically mildest mechanism of raising the valence of an oxide by its anodic dehydration (reaction (12)).

This is entirely valid at alkali concentrations to about 5.0 N. As the strength of the solution increases, the derivative dE/dn decreases within the range $n > 1.5$ and at the oxidization limit the mass of a nickel-oxide electrode may reach the composition $NiO_{1.80}$. It must be emphasized, however, that this evidently higher stability of higher-valence nickel oxides is related to their different nature and brought about by the participating oxide of the alkali metal, which the electrode in such a solution absorbs during charge and releases during discharge in an amount of up to four atoms per ten nickel atoms [13]. The new crystalline phase forming in such a process is denoted as γ-NiOOH [13, 15, 17]. Actually, of course, we ought to be concerned here not with an individual nickel oxide but with the partial formation of binary oxides (nickelates), i.e., salts whose dissociation in a strong alkali is difficult. An evaluation of the thermodynamic characteristics of these complex compounds is beyond the scope of this report.

In concluding the survey of reactions and compounds listed in Table 1, we note that the existence and the properties of the hypothetical NiO_4^{2-} ion suggested in [6] are also not plausible, inasmuch as the experiments [4] on which they are based have been proven incorrect [23].

And so our analysis indicates that, instead of the series of hypothetical phases whose participation in the action of a nickel-oxide electrode has been regarded self-evident or provable on the basis of various misinterpretations, in not too highly concentrated alkali solutions we actually find only two such phases in existence: $Ni(OH)_2$ and β-NiOOH. The electrode reaction (12) involving them and proceeding along the flat segment of either the charge curve or the discharge curve is, moreover, the only electrochemical phase transformation which occurs in a nickel-oxide electrode.

Assuming $E_{12} = 0.505 + 0.059pOH \pm 0.020$ V as the equilibrium potential of this reaction, we find the difference between the standard values listed for the free energy of β-NiOOH and $Ni(OH)_2$ formation respectively (from the elements Ni, O, H) to be 30.7 ± 0.5 kcal/mole. With ΔG^{298} for $Ni(OH)_2$ taken as the arithmetic mean of values characterizing a freshly formed hydrate (−106.84 kcal/mole) and an aged hydrate (−109.55 kcal/mole) [26], under conditions of a working nickel-oxide electrode, we obtain $\Delta G^{298} = -77.5 \pm 0.5$ kcal/mole for β-NiOOH.

Mixed oxide, if it forms on a nickel-oxide electrode (according to [2], the only report mentioning this), forms only in the presence of both $Ni(OH)_2$ and β-NiOOH together, i.e., at the potential E_{12} and then extremely slowly. The free energy of mixed-oxide formation from $Ni(OH)_2$ and β-NiOOH is thus, if not positive, slightly below zero and this compound may, therefore, be regarded only as one among the crystalline forms of existence of hydrous oxide and hydroxide in equilibrium, which energetically is approximately equivalent to their coexistence as separate phases. Not having its own characteristic formation potential, Ni_3O_4 cannot play any independent role in the series of electrochemical transformations of nickel oxides. This distinguishes it from Fe_3O_4, whose formation on an iron electrode of a similar alkaline battery, e.g., is easily revealed by x-ray examination as well as by the characteristic potential delay on the charge curve [27].

It follows that the range of electrochemical stability of the $Ni(OH)_2$ phase extends over more than 1.2 V, from the potential of its formation from metallic nickel ($E \sim -0.71 + 0.059pOH$, according to the accepted value of ΔG^{298} [26]) to the potential E_{12}, the latter being 0.1 V more positive than the equilibrium potential of an oxygen electrode. A rise of the potential within this range will, of course, make the phase become somewhat nonstoichiometric. The average equilibrium valence of the metal will never exceed 2.2, i.e., that corresponding to $NiO_{1.10}$ (Fig. 1), however, even at the instant when transformation to β-NiOOH occurs. Considering the earlier-mentioned loss of contact between the current feeder and individual fragments of unreduced β-NiOOH, this average equilibrium valence will probably be still lower.

In turn, the β-NiOOH phase becomes electrochemically stable only at the potential E_{12} and remains so up to a potential at least still 0.2 V more positive so that the formal valence of the metal here can reduce to about 3.2 (corresponding to $NiO_{1.6}$ in Fig. 1).

In order to clearly relate the significance of the foregoing statements to corrosion systems, we will now consider acid solutions and use the earlier-found value of the free energy of β-NiOOH formation for calculating the potential of the reaction

$$NiO+H_2O \rightleftharpoons \beta\text{-}NiOOH+H^+ + e,\ E = \sim +1.3 - 0.059\ pH\ V. \quad (13)$$

The potential of NiO to Ni_2O_3 oxidation cannot be much lower than this, or it would mean a substantial decrease in the energy of the 1.5-valent oxide with the removal of the hydrate water and thus a tendency of β-NiOOH in nickel-oxide electrode to dehydrate, which nobody has ever noted.

Consequently, 1.3 V is approximately the potential below which a phase with the lower-valence nickel oxide in a solution with pH = 0 cannot be at all transformed electrochemically to a phase with the higher-valence oxide (refer to the earlier discussion of Ni_3O_4) and this is entirely consistent with the well-known stability of NiO in air [28].

But then, contrary to customary assumptions [29-31], neither a 1.5-valent nickel oxide nor a mixed nickel oxide could be responsible for the peculiar trend of the anodic polarization curves for nickel within the ranges of passivation and stable passive state. The hypothesis in [32] is obviously closer to reality, attributing the passivating characteristics to a nonstoichiometric lower-valence oxide, but a proviso is in order here that this oxide must, in all probability, have the structure of a lower-valence oxide with the ratio O:Ni < 1.1 at least at the beginning of the passivation range. Conversely, the peculiar anodic behavior of oxygen on nickel and the attendant overpassivation effects must, it seems, be regarded first of all with a consideration of the characteristics of this 1.5-valent nonstoichiometric nickel oxide rather than of the hypothetical higher-valence nickel oxide [24, 25].

CONCLUSIONS

1. The thermodynamic characteristics of higher-valence nickel oxides, as listed in Latimer's handbook [7] and in Pourbaix' charts [6] or in other handbooks using their values, are incorrect and unusable. Only the existence of 1.5-valent phase oxides and their participation in electrode processes are indisputable, but their standard formation energy (–77.5 ± 0.5 kcal/mole for β-NiOOH) is much more positive than listed.

2. In view of our analysis, all hypotheses regarding the participation of higher-valence nickel oxides in passivation, overpassivation, and oxygen evolution at a nickel anode ought to be reviewed.

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CORROSION CRACKING AND THE DISLOCATION SUBSTRUCTURE OF AUSTENITIC IRON–CHROMIUM–NICKEL ALLOYS

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A study was made concerning the dislocation substructure of austenitic Fe–Cr–Ni alloys (16-20% Cr, 10-60% Ni) with various degrees of proneness to chloridic transcrystalline corrosion cracking. When deformed under tension to a strain level within 8-10%, all these materials, and among them the corrosion-resistant nickel alloy, exhibited a coplanar (tabular) distribution of dislocations. A change in the dislocation structure of austenitic steel (16% Cr + 15% Ni) affected the degree of proneness to corrosion cracking, but the proneness remained with every substructure. An electron-microscope examination has revealed that during the initial stage of corrosion cracking there form micropits with pointed protrusions on the surface. The corrosion products on the surface trace of a crack are distributed continuously throughout and differ distinctly from the film on the remaining surface as well as from the corrosion products inside a crack. A probable mechanism is described by which dislocation arrays affect the crack initiation and development.

Electron-microscopic examinations made during the last few years have revealed that dislocation arrays constitute regions where transcrystalline cracks are initiated and develop during corrosion cracking. A coplanar (tabular) dislocation substructure (with dislocations distributed in parallel slip planes) has been found characteristic of copper alloys and grade 8-10 austenitic chromium–nickel steel, which are prone to transcrystalline corrosion cracking [1-3]. The object of this study was: 1) to correlate the dislocation substructure with the proneness to corrosion cracking of Fe–Cr–Ni alloys of various compositions, and 2) to establish whether there is any relation between the dislocation structure and resistance to corrosion cracking in any given material.

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