

Phase transformations during electrochemical incorporation of lithium in intermetallic compounds of aluminum

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Comparative study of the regularities of the reaction and specific features of phase formation during electrochemical incorporation of lithium from propylene carbonate solutions in intermetallic aluminum-based compounds (CuAl_2 , Mg_2Al_3 , and NiAl) and pure metals (Al, Cu, Mg, and Ni) was performed. The initial stage of the process was shown to be dissolution of lithium in the solid phase limited by diffusion for all studied substrates. Trace amounts of lithium-containing by-products were detected in NiAl , Ni, and Cu samples. The subsequent change in the limiting stage is related to the beginning of formation of a new phase: metallic lithium (on Mg_2Al_3 , NiAl , Mg, Ni, and Cu) or LiAl (on Al and CuAl_2 cathodes). In the latter case, the solid-phase substitution occurs, which is formally described by the equation: $\text{CuAl}_2 + 2\text{Li}^+ + 2e \rightarrow 2\text{LiAl} + \text{Cu}$. Thus, the specific features of phase formation on the CuAl_2 electrode correspond to the highest (among three intermetallides studied) concentration of Al atoms in the crystal lattice of the compound.

Key words: intermetallic compounds, electrochemical incorporation, phase formation, lithium, aluminum.

Electrochemical incorporation of metals in electrodes resulting in the formation of intermetallic compounds (IMC) is a special type of heterogeneous solid-phase reactions, which are controlled by the potential and include several successive stages. The kinetics of the latter is determined by the nature of the incorporated metal and metal support, the composition and nature of the electrolyte solution, surface properties of the electrode, and temperature.¹ The theoretical significance of studying the kinetic regularities and mechanism of these reactions is combined with the possibilities of practical use of this phenomenon for preparing active electrode materials of primary and secondary lithium current sources.²⁻⁴ It is very promising to use electrochemical incorporation for synthesis of surface layers of new, including metastable, IMC,¹ which often possess properties that are weakly pronounced or altogether absent for the starting metals.

In the majority of works devoted to the elucidation of the kinetics and phase formation during similar processes, the electrochemical incorporation of alkali metals, in particular lithium, in one-component metallic electrodes was studied.^{1,2,5-10} The use of two-component alloys instead of pure metals as the starting matrix may change the kinetics of cathode incorporation and subsequent selective anode dissolution of lithium, and the phase composition and morphological stability of the surface layer of lithium-containing compounds.⁹⁻¹² The effect of the nature of the doping component and composition of the starting metallic electrode on the

electrochemical process and behavior of the lithium-containing alloy in non-aqueous solutions was studied in most detail for two structural types of aluminum alloys: aluminum-based solid solutions and biphasic systems.^{9,10} In both cases, lithium adsorption by the aluminum component of the alloys occurs mainly during incorporation.^{9,10} At the same time, both the possibility of electrochemical incorporation of lithium in alloys, which are intermetallic compounds and differ from the starting metals by the type of the crystal lattice, and specific features of the kinetics and phase formation during this process remain unclear.

The purpose of this work was to elucidate the possibility of electrochemical incorporation of lithium in aluminum-based IMC CuAl_2 , Mg_2Al_3 , and NiAl and to study the kinetics and phase formation during the reactions.

Experimental

Experiments were carried out on disk electrodes with an effective surface area of 0.2 cm^2 prepared from CuAl_2 , Mg_2Al_3 , NiAl , Al (99.9%), Cu (99.9%), Mg (99.9%), and Ni (99.9%). Intermetallides were obtained by fusion of pure metals with flux (CuAl_2) or in an inert atmosphere (Mg_2Al_3 , NiAl). The structure of IMC was established by X-ray powder analysis (XPA).

Prior to each experiment, the working surface of the electrodes was treated by fine-grained (00) emery paper, washed with distilled water, and degreased by refluxing in EtOH for 1 min.

A 0.5 M LiClO_4 solution in propylene carbonate (PC) was used as the electrolyte. Prior to preparation of the solution, LiClO_4 (chemically pure) was dried with successive temperature increases from 100 °C to 200 °C to remove crystallization water. High-temperature drying was performed under reduced pressure. Moisture was removed from PC by keeping it above molecular sieves 4 Å. The content of water in the electrolyte prepared was not higher than 0.01 vol. %.

Polarization measurements were carried out in a glass three-electrode cell with unseparated cathodic and anodic areas. The working and accessory (Pt) electrodes were molded in fluoroplastic holders and hermetically inserted into the cell. A saturated Ag/AgCl electrode connected with the cell by an electrolytic bridge filled with a working solution served as the reference electrode. All potentials are presented relative to the reference lithium electrode.

A PI 50.1.1 potentiostat was used to maintain the specified current values, and changes in the working electrode potential in time were detected by a KSP-4 recording potentiometer. The specified current values were monitored by an Shch-4313 voltammeter.

Since the kinetics of cathode incorporation is very sensitive to the state of the electrode surface,² the reproducibility of values of the currentless electrode potential and the character of its change in time was used as the criterion of standardization of its state. Lithium was electrochemically incorporated only in the electrodes, whose measured values of currentless potentials were within the admitted range: 2.58–2.60 (CuAl_2), 2.28–2.30 (Mg_2Al_3), 2.74–2.76 (NiAl), 2.34–2.36 (Al), 3.00–3.02 (Cu), 1.48–1.50 (Mg), and 2.72–2.74 (Ni) V.

X-ray powder analysis of electrode surfaces before and after lithium incorporation was carried out on a DRON-2.0 diffractometer using $\text{Cu-K}\alpha$ radiation, $\lambda_{\alpha} = 0.154178$ nm, the Bragg–Brentano method. After incorporation, the electrodes were analyzed in a hermetic polyethylene (PE) shell, which restricted accessibility of moisture (followed by sifting diffraction maxima corresponding to PE). The published data¹³ and our calculations of X-ray patterns from parameters of unit cells and atomic coordinates were used for interpretation of XPA results.

The microhardness of the metals was determined on a PMT-3 instrument with loading on the diamond pyramid of 20–50 g using a standard procedure.

Results and Discussion

Comparative analysis of the regularities of the electrochemical incorporation (deposition) of lithium on cathodes of pure metals and their IMC was performed

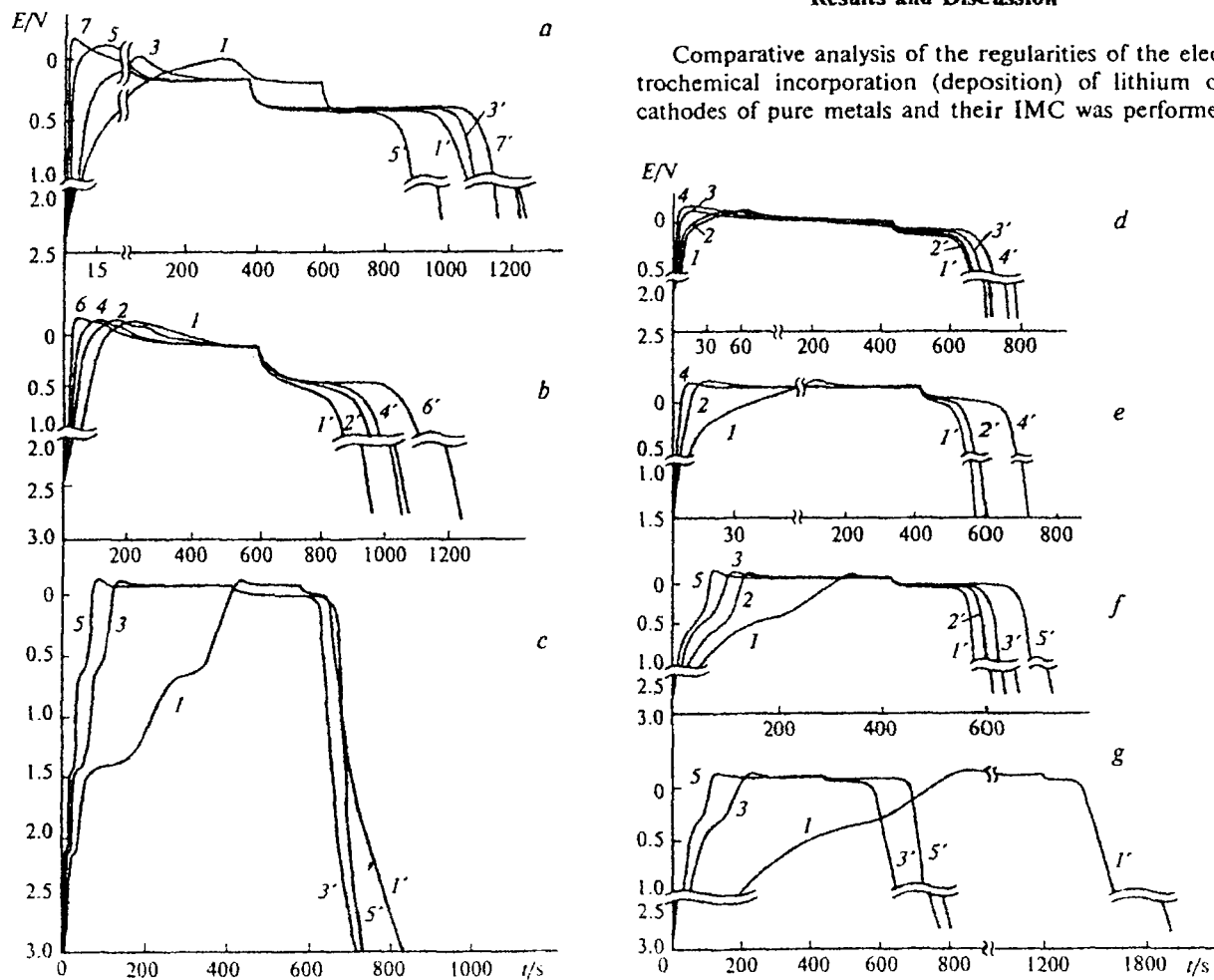


Fig. 1. Cathodic (1–7) and subsequent anodic (1'–7') chronopotentiograms of Al (a), CuAl_2 (b), Cu (c), Mg_2Al_3 (d), Mg (e), NiAl (f), and Ni (g) electrodes in 0.5 M LiClO_4 in PC at current densities: –0.2 (1), –0.3 (2), –0.4 (3), –0.5 (4), –0.6 (5), –0.7 (6), –0.8 (7), 0.1 (1'), 0.15 (2'), 0.2 (3'), 0.25 (4'), 0.3 (5'), 0.35 (6'), and 0.4 (7') mA cm^{-2} .

from the results of cathodic, anodic, and currentless chronopotentiometry. The corresponding potential delays in the cathodic and subsequent anodic (currentless) chronopotentiograms (CPG) of the electrode can be related, according to the Gibbs phase rule,^{1,14} to the existence of an equilibrium biphasic system on the electrode surface. A similar procedure of detection of phase components in metal alloys is widely used for studying phenomena of electrochemical incorporation.^{1,6,7}

The chronopotentiograms of the intermetallic and pure metal electrodes (Fig. 1, *a–g*) are qualitatively similar: after the cathodic current is switched on, the potential is more or less monotonically shifted to negative values, the overvoltage reaches a maximum, and the potential values are stabilized. Taking into account the data obtained for the aluminum electrode,¹⁵ we can assume that the extremum on the galvanostatic dependence (time (*t*) dependence of the potential (*E*) is related to the formation of a new phase accompanied by a change in the type of the controlling stage. In this case, the values of the extremum coordinates (E_{\max} and t_{\max}) and their sensitivity to the current density are important parameters of the incorporation process. An increase in the current density results in faster achievement of the maximum and an increase in the overvoltage of the process (see Fig. 1). The anodic CPG of all electrodes studied exhibit a delay after which the potential decreases to positive values and approaches the corresponding currentless electrode potential (see Fig. 1, *a–g*, curves 1'–7'). The time of the potential delay increases as the cathode current density (i_c) increases (at the same time of cathode polarization).

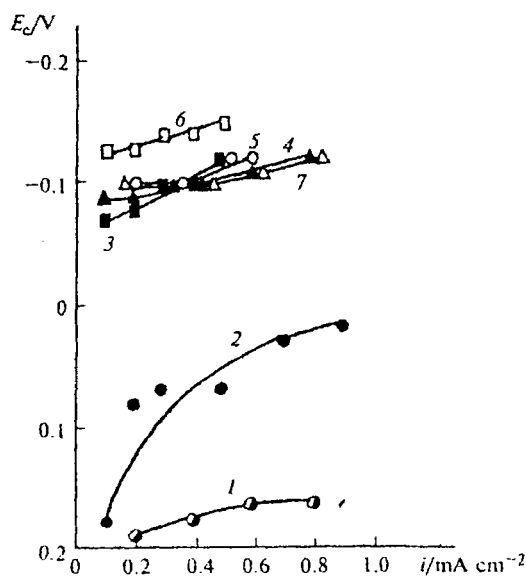


Fig. 2. Dependences of the post-extreme regions of cathodic chronopotentiograms (E_c) on the cathodic current density. Composition of the support: 1, Al; 2, CuAl_2 ; 3, Mg_2Al_3 ; 4, NiAl; 5, Cu; 6, Mg; and 7, Ni.

However, the quantitative similarity of the general form of the $E(t)$ dependences of the supports studied cannot prove that the phase transformations are also similar. The difference between the processes occurring on the electrodes is indicated, first of all, by the difference in the number and potentials of delays in the cathodic and subsequent anodic CPG (see Fig. 1, *a–g*) and in the values of potentials of the post-extremum regions (E_c) of the cathodic CPG (Figs. 1 and 2).

For example, for Al–(Li) and CuAl_2 –(Li), the values of potential delays in the anodic CPG are 0.5–0.4 V, while for all other electrodes, they are 0.03–0.07 V (depending on the i_c and i_a values) (see Fig. 1, *a–g*, curves 1'–7'). The potentials of the post-extremum regions of the cathodic $E(t)$ dependences for the supports of Mg_2Al_3 , NiAl, Mg, and Ni are shifted to the negative region, whereas those for the Al and CuAl_2 supports are shifted to the positive region relative to the currentless potential of the Li/Li⁺ electrode (see Fig. 2). In addition, for the Al and CuAl_2 cathodes, the dependences of E_{\max} on i_c are linear (correlation coefficients $r = 0.9965$ and $r = 0.9933$, respectively), which is not characteristic of the other systems under study ($r < 0.98$). The values of t_{\max} for the CuAl_2 and Al cathodes are close at different current densities, whereas for Mg_2Al_3 and Mg, the extremum of the potential is achieved more rapidly, and that for NiAl, Ni, and Cu, more slowly than that for Al (see Fig. 1, *a–g*, curves 1–7).

The results obtained suggest that the nature of the lithium-containing phases formed in the cathode polarization (and then dissolved on the anode) on CuAl_2 and that of the other intermetallic supports are different.

Since the possibility of successive phase formation during electrochemical incorporation cannot be ruled out,^{1,9} the quantitative distribution of lithium between different phases can change with time. The inflection points observed in the initial regions of the cathodic CPG are evidence for the specific features of phase formation during the electrochemical incorporation of lithium in some electrodes. For example, the NiAl and Ni cathodes are characterized by a short potential delay within 0.5–0.4 V (depending on the i_c value) (see Fig. 1, *f*, curves 1–3, and 5; Fig. 1, *g*, curves 1, 3, and 5). The pre-extremum region of the cathodic CPG of the copper electrode contains three potential delays near $E = 2.15$, 1.45, and 0.75 V (see Fig. 1, *c*, curves 1, 3, and 5), which are absent in the cathodic dependences of E on t for all other supports. However, the existence of minor amounts of the primarily formed phase components cannot be detected in the subsequent analysis of the anodic CPG due to their small specific fraction or complete transformation into more lithium-enriched compounds.

To find lithium-containing phases formed at the initial stages of electrochemical incorporation, we studied the curves of decay of the currentless potential of the electrodes after switching-off the cathode current at different moments (Fig. 3, *a–g*). The potential delay in

the region of 0.9–0.8 V is observed in the currentless curves of the $E(t)$ dependence for the NiAl–(Li) and Ni–(Li) electrodes when the cathode polarization is switched off after detection of the inflection point in the region of $E = 0.5$ –0.4 V and after passing through the maximum of the cathodic CPG (see Fig. 3, *f*, curves 2 and 3; Fig. 3, *g*, curves 2 and 3). This delay was not detected in the anodic CPG of these electrodes (see Fig. 1, *f*, curves 1'–3', and 5'; Fig. 1, *g*, curves 1', 3', and 5') and can correspond to dissolution of lithium from an intermediate intermetallide phase formed during the cathode incorporation of lithium in NiAl and Ni. For the Mg_2Al_3 support, when the cathode current is switched off and E_{\max} is achieved, the curves of currentless potential decay exhibit a slight inflection point near $E = 0.4$ V (see Fig. 3, *d*, curve 2), which also was not detected in the corresponding anodic CPG (see Fig. 1, *d*, curves 1'–4'). The potential delay of ~1 V observed¹⁶ in the currentless $E(t)$ dependences of the Al–(Li) electrode (see Fig. 3, *a*, curves 2 and 3) is related to dissolution of lithium from the metastable δ' -phase of Al_3Li .

The obtained currentless CPG of the electrodes confirm the previous assumption on the relation of the extremum on the cathodic curves of the $E(t)$ dependence of the electrodes studied to the beginning of crystallization of a new phase: in the case where the time of cathode polarization exceeds t_{\max} , a new delay of the potential appears

in the currentless CPG (see Fig. 3, *a*–*g*). For the $CuAl_2$ and Al cathodes, the interrelation of the overvoltage in the maximum (η_{\max}) of the cathodic CPG and i_c is well described by the equation

$$\ln i_c = k_1 \eta_{\max}^{-2} + k_2, \quad (1)$$

where $k_1 = -0.34 \pm 0.03$ (V^2), $k_2 = 0.89 \pm 0.05$ for Al (correlation coefficient $r = 0.9991$) and $k_1 = -3.88 \pm 0.44$ (V^2), $k_2 = 7.71 \pm 0.98$ for $CuAl_2$ ($r = 0.9901$) (i_c was expressed in $mA\ cm^{-2}$). Dependence (1) corresponds to the Volmer equation¹⁶ and indicates the retarded stage of formation of three-dimensional nuclei of the new phase (LiAl) at the moment when a maximum in CPG is achieved.

Taking into account the published data,^{1–8,17} we can deduce that the similarity of the cathodic CPG of all supports studied (the extremum of the potential followed by its stabilization) is due to the transition from diffusion control (diffusion of lithium in the solid phase) to crystallization-chemical control. At the same time, the nature of the phase (during the formation of which the limiting stage changes) differs for two groups of electrodes: (1) Al and $CuAl_2$ and (2) all other supports. The difference in the character of processes occurring on two groups of electrodes is also confirmed by the comparison of the state of their surfaces after the end of the prolonged cathode incorporation of lithium. An increase in the cathode volume and cracking of

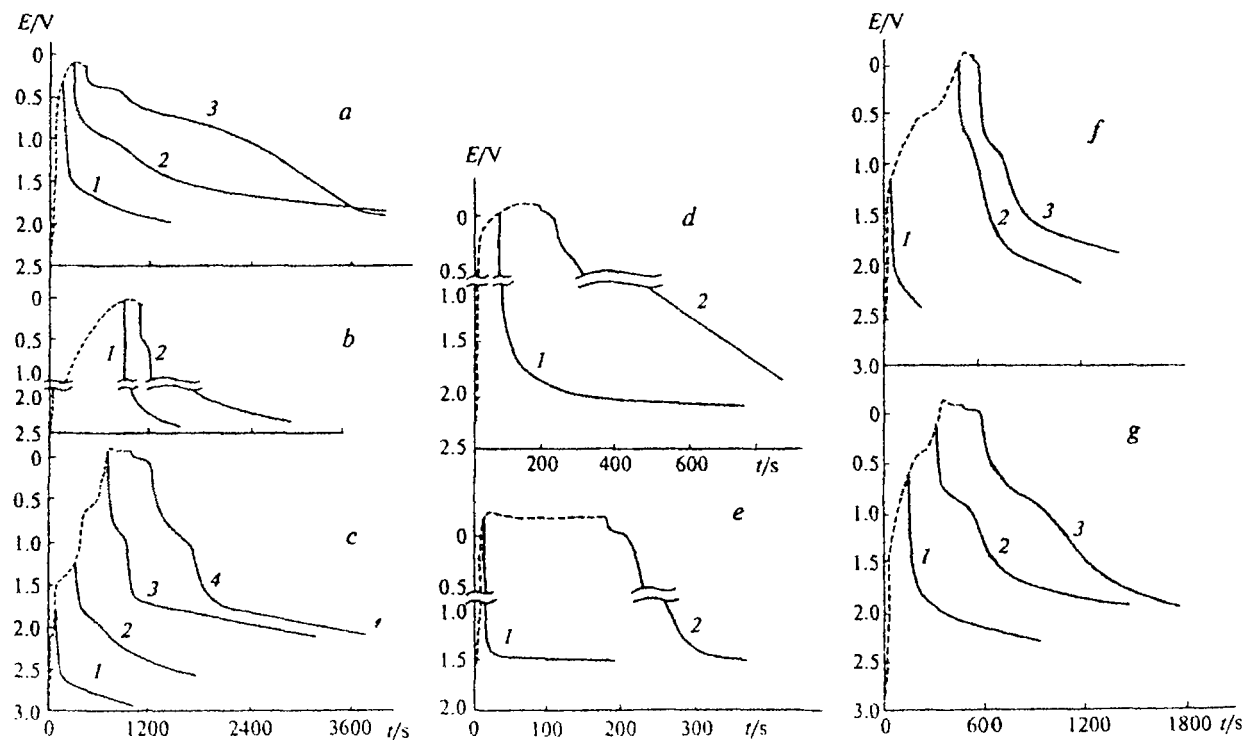
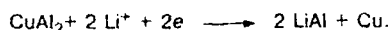


Fig. 3. Curves of decay of the currentless potential of electrodes after switching off the cathodic current at different moments. The corresponding cathodic chronopotentiogram at $i_c = 0.1\ mA\ cm^{-2}$ (*a*–*d*, *f*) and $0.3\ (e, g)\ mA\ cm^{-2}$ are shown by the dotted line. Composition of the starting electrode: Al (*a*), $CuAl_2$ (*b*), Cu (*c*), Mg_2Al_3 (*d*), Mg (*e*), NiAl (*f*), and Ni (*g*).

the surface layer, which are typical of lithium incorporation deep into the matrix to form a new low-density phase,^{2,8,10} was observed on the Al—(Li) and CuAl₂—(Li) electrodes. For all other electrodes, the formation of a surface precipitate, which interacts actively with the atmospheric components, was observed.

The comparison of the number of values of the delay potentials detected in the cathodic, anodic, and currentless curves of the $E(t)$ dependence of the electrodes studied supplements the known published data on the electrochemical behavior of magnesium,¹⁸ nickel,⁷ and, especially, copper¹⁹ during cathode incorporation of lithium. In particular, we can speak of the formation of at least one intermediate compound during lithium incorporation in Ni and two compounds during lithium incorporation in Cu.

Thus, the results of chronopotentiometric and microscopic studies indicate that the formation of the solid solution of lithium (α -phase) on the Al and CuAl₂ electrodes changes during cathode incorporation by the formation of IMC LiAl (β -phase), which moves deep into the matrix. The copper formation on the intermetallide cathode can be expected according to the formal reaction equation:



The diffusion-controlled lithium incorporation (to form a solid solution or minor amounts of IMC* containing no aluminum) occurs primarily in the solid phase on pure Mg, Ni, Cu, and IMC Mg₂Al₃ and NiAl. In this case, the change in the nature of the controlling stage is related to supersaturation of the surface layer of the electrode with lithium and the beginning of electrodeposition of its phase layer.

These conclusions are confirmed to a considerable extent by the results of XPA performed on the Al and intermetallide electrodes subjected to prolonged (from 3 to 8 h) cathode polarization in the galvanic- and potentiostatic regimes ($Q_c = 25\text{--}35 \text{ C cm}^{-2}$). In all X-ray patterns, the most pronounced reflections corresponded to the unchanged main phase of the electrode, which is the result of an insignificant thickness of the surface layer formed during lithium incorporation. For the pure aluminum electrode, intense and distinct lines of the β -phase (LiAl) were detected after incorporation. They indicate its good crystallization and a high concentration in the surface layer. In the case of the CuAl₂ electrode, only traces of the β -phase and Cu displaced from the starting IMC during the electrode process were observed (see above). This can be related to an insignificant thickness of the unchanged layer and poor crystallization of the phases formed.

* Most likely, an insignificant amount of the β -phase (LiAl) is formed on the Mg₂Al₃ cathode during the reaction. This is indicated by the potential decay at values close to the potential corresponding to equilibrium $\alpha\text{-phase} + n\text{Li}^+ + ne = \beta\text{-phase}$ in the currentless CPG of the electrode. (see Fig. 3, *d*, curve 2)

Table 1. Values of some parameters indirectly characterizing structural energetic properties of crystal lattices of IMC and Al

Phase	ΔH_f^o ^a kJ mol ⁻¹	ΔH_{at}^{sp}	Micro- hard- ness /kg mm ⁻²	V_{sp} ^b /Å ³	W_{Al} ^c vol.%	at.%
Al	0	326.4	13.2	16.6	74.05	100
CuAl ₂	-39.8	343.6	165	14.9	54.93	66.7
Mg ₂ Al ₃	-41.9	263.3	93.0	19.1	38.54	60.0
NiAl	-58.8	407.5	297	13.8	51.20	50.0

^a The values for IMC were calculated according to the Hess law from enthalpies of formation²⁰ and enthalpies of atomization of simple substances.²¹

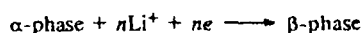
^b $V_{sp} = V/Z$, where V is the unit cell volume, and Z is the overall number of atoms in the cell.

^c The metrics and composition of the unit cell and the system of metal radii were used in the calculation.²²

It is difficult to reveal the Li phase by XPA because of its low X-ray scattering factor. Nevertheless, two most pronounced reflections of Li can be satisfactorily observed for the NiAl electrode, which agrees with the results of chronopotentiometric studies. Traces of lithium are also probable in the case of Mg₂Al₃; in this case, interpretation of X-ray patterns is impeded by the superposition of reflections of the starting and forming phases.

No other intermetallides from the Al—Cu, Al—Mg, and Al—Ni systems and those involving Li were observed by XPA. The formation of LiOH, Li₃N, or Li₂CO₃ was also suppressed.

It should be mentioned in conclusion that comparison of the structural and surface parameters of the three IMC of aluminum studied (Table 1) does not explain unambiguously the formation of the β -phase only in the case of one of the IMC (CuAl₂). The values of enthalpies of formation (ΔH_f^o) and specific enthalpies of atomization (ΔH_{at}^{sp}) of the intermetallides (parameters of thermodynamic stability of compounds), their microhardness (indirect characteristic of the work on the formation of a new phase²³), and specific volume (V_{sp}) per metal atom in the crystal lattice indicate that CuAl₂ occupies an intermediate position between NiAl and Mg₂Al₃. The highest (compared to other IMC) concentration of aluminum atoms (W_{Al} , at.%) and the volume fraction occupied by them in the crystal lattice of the compound (W_{Al} , vol.%) (see Table 1) are the only factors favoring the transformation



precisely on the CuAl₂ cathode. The low molar fraction (specific volume) of aluminum is most likely the main factor impeding the nucleation and growth of the β -phase (most probably, due to kinetic reasons), whereas the high values of strength parameters of the crystal lattice (ΔH_f^o , ΔH_{at}^{sp}) also play an important role for NiAl. Under these conditions, the formation of the phase of

pure Li on the cathode surface occurs with a lower overvoltage than the formation of β -LiAl.

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