



## CHARACTERISTICS OF IRON-LITHIUM PHOSPHATE COATINGS

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**Annotation:** Galvanic coatings have a clear crystalline structure, so the process of cathodic restoration of metals is called electrocrystallization. Many studies show that the denser and smaller the structure of the coating, the higher their properties. The kinetics of the electrocrystallization process is expressed by two main parameters: the rate of formation of crystallization centers or crystals, which occur on one surface at the same time, and the linear rate of their growth. The factor that determines the rate of formation of crystals during crystallization of the solid phase of the solution is the degree of its supersaturation. Each crystal body consists of many crystals called crystallites or grains with linear dimensions of  $10^{-5}$ - $10^{-3}$ , and each grain - crystallite is arranged in an irregular shape that prevents the growth of neighboring crystals is a crystal that has

**Key words:** Galvanic coatings, electrocrystallization, crystallite, phosphating, polyanionic compounds, olivine, degreasing, neutralization, disassembly.

Physical and mechanical properties also depend on the texture of the metal coating. Texture refers to the dominance of crystals in one or more directions. Before the formation of its texture, the metal consists of formed crystals of the same type, but oriented in different ways in space. When tissues are formed, the orientation of many crystals is parallel to some direction called the axis of the tissue.

The directional effect of the parent metal structure is also lost when using high current densities, as large amounts of randomly oriented crystals are formed or the specific orientation of the deposited metal is created. For example, zinc deposition on a polished brass plate is first a small crystal deposit with randomly located crystals, then a large number of zinc crystals grow along the common direction axis.

At the same time, to obtain the isotropic properties of polycrystalline coatings, it is not uncommon to eliminate the texture with a special treatment. Currently, the value of textures is evident due to the different requirements for the properties of coatings obtained by electrochemical means. Cathode materials of iron-lithium phosphate coatings must be capable of reversible insertion/extraction of  $\text{Li}^+$ . The  $\text{Li}^+$  extraction/acceptance potentials of these compounds should be lower than the oxygen release potentials to ensure the stability of aqueous electrolytes. However, to increase the energy density, this potential must be increased. With these considerations, various cathode materials, including oxides ( $\text{LiMn}_2\text{O}_4$ ,  $\text{MnO}_2$ ,  $\text{LiCoO}_2$ ,  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ ), polyanionic compounds ( $\text{LiFePO}_4$ ,  $\text{FePO}_4$ ,  $\text{LiMnPO}_4$  etc.).

Cathode materials at various stages have shown limited specific capacity and significant power loss during battery cycling. Studies have shown that the limitation of specific capacity is due to (1) incorporation of  $\text{H}^+$  into the structure, (2) exchange during the  $\text{Li}^+/\text{H}^+$  cycle, (3) water penetration into the structure, and (4) dissolution of active materials in the water

electrolyte, related to Many studies have addressed these issues by modifying the cathodes with additives or additives and by coating the material/electrolyte interface with all kinds of coatings or by changing the electrolyte composition.

The first cell prototype  $\text{LiMn}_2\text{O}_4$  with a cathode showed significant improvements, with an average voltage of 1.5 V and an energy density of 75 W/s/kg, which could compete with nickel-cadmium and lead-acid batteries. It is important to choose an electrolyte with a specific pH value to ensure efficient charging of cathode materials. Because in the process of removing lithium ions from  $\text{LiMn}_2\text{O}_4$ ,  $\text{O}_2$  can be released at high pH values, which worsens the performance of the cycle. Further research on the effect of pH on the electrochemical reactions of  $\text{LiMn}_2\text{O}_4$  showed that the introduction of  $\text{H}^+$  at low pH can be the exchange of  $\text{H}^+$  and  $\text{Li}^+/\text{H}^+$ , so the aqueous  $\text{LiMn}_2\text{O}_4$  cathodes reliable operation and it is very important to maintain a certain pH value of the electrolyte.

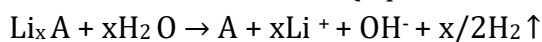
$\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  (NCM) was introduced as the cathode material as an alternative perspective. The stability of NCM depends on solution pH. It was found that at pH 7 and 9, negative reactions occur in the electrolyte,  $\text{H}^+$  is introduced into the NCM electrode, and this material works stably at pH 11-13 synthesized NCM with polypyrrole coating and studied its electrochemical properties using  $\text{LiV}_3\text{O}_8$  as anode and 5m  $\text{LiNO}_3$  as electrolyte. NCM polypyrrole-coated NCM exhibits 70 ma/g in the first discharge cycle, maintaining 55 ma/g after 150 cycles.

For coating,  $\text{LiFePO}_4$  uses olivine as a cathode. After a series of electrochemical tests, scientists found that the electrochemical oxidation of  $\text{LiFePO}_4$  with the formation of  $\text{FePO}_4$  is similar to anhydrous systems.

$\text{FePO}_4$ , similar to olivine, was investigated in 2010 as a potential cathode for aqueous Li solutions. Based on the XRD analysis, it was found that  $\text{FePO}_4$  occurred with the formation of olivine  $\text{LiFePO}_4$ . This shows that the redox reaction mechanism for  $\text{FePO}_4$  in aqueous  $\text{LiOH}$  electrolyte is the same as in anhydrous  $\text{LiOH}$  electrolyte. The  $\text{FePO}_4$  electrode produces a capacity of 65 ma-h/g with an average of 0.5 V (relative to the zinc anode) over the first discharge cycle.

The following substances were proposed as anode materials for iron-lithium phosphate coatings: oxides ( $\text{VO}_2(\text{B})$ , spinel ( $\text{Li}_2\text{Mn}_2\text{O}_4$ ), layered  $\gamma\text{-LiV}_3\text{O}_8$ , paramorph  $\text{VO}_2$ ,  $\text{V}_2\text{O}_5$  and anatase  $\text{TiO}_2$ ), superconductors such as polyanionic compounds (pyrophosphate  $\text{TiP}_2\text{O}_7$  and  $\text{LiTi}_2(\text{PO}_4)_3$ ), whose redox reactions occur near the potential of hydrogen release.

Based on preliminary studies, most anode materials degrade significantly during the electrochemical reaction, limiting their practical potential compared to conventional lithium-ion batteries. Research on anode materials has revealed the reasons for maintaining a low potential: (1) dissolution of active compounds; (2) irreversible structural changes, possibly proton insertion, and (3) spontaneous extraction reactions from lithium substituents leading to the formation of  $\text{LiOH}$  (aqueous solution) and hydrogen



can be significantly improved by precisely controlled pH, type and concentration of lithium salt, residual  $\text{O}_2$  and surface coating of active materials using electrolytes.

Initially,  $\gamma\text{-LiV}_3\text{O}_8$  was introduced as a possible cathode material for conventional solutions, aqueous LEADA as an anode material was later proposed by Kohler et al.  $\gamma\text{-LiV}_3\text{O}_8$  electrochemical tests show the thermodynamic stability of  $\text{Li}_{1+x}\text{V}_3\text{O}_8$ , as the potential of the redox reaction (-100 mV against the standard hydrogen electrode) is hydrogen separation at

pH 6.2 (-366 mV against the standard hydrogen electrode) above potential. Nevertheless, the material has low performance (capacity 45÷90 ma/g, 230 ma/g in organic electrolyte and only 25-40% of the initial capacity is retained after 100 cycles). In 2007-2008, Wang studied the electrochemical behavior of  $\text{LiV}_3\text{O}_8$  anode with various cathode materials such as spinel, layered oxides, polyanionic compounds listed above. He also reported a capacity of 70 ma ch h/g in aqueous systems, which was below 50% after 100 cycles in a neutral environment. Next, the reason for the low performance of g- $\text{LiV}_3\text{O}_8$  it was found that the crystal structure deteriorates during the cycle.

Presented  $\text{LiMn}_2\text{O}_4$  spinel as an anodic material in aqueous LIADA. It was shown that the discharge profile of  $\text{LiMn}_2\text{O}_4$  has a voltage plateau of-100 mV (relative to a standard hydrogen electrode). The cycling properties of these anodes have been improved by several approaches. In order to improve the stability of  $\text{LiTi}_2(\text{PO}_4)_3$ , it has been shown that it is important to maintain a neutral pH, which eliminates water decomposition.

The following technological system was developed for obtaining iron-lithium phosphate coatings:

Table 1

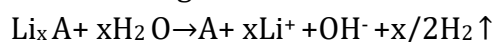
N	Process name and order	Device name	Substances	Concentration, g/l	Temperature, °C	Current-density A/dm <sup>2</sup>	Time, minute	Indicators
1.	Loading the parts into the drum for phosphating	A vending machine AX -42					10	
2.	Chemical degreasing in the 1st bath	Hot tub	NaOH KOH $\text{Na}_2\text{CO}_3$ $\text{Na}_3\text{PO}_4$ Liquid bottle	60-100 30-40 15-20 2-4	60-90	6-8	7-10	
3.	Chemical degreasing in the 2nd bath	Hot tub	NaOH KOH $\text{Na}_2\text{CO}_3$ $\text{Na}_3\text{PO}_4$ Liquid bottle	60-100 30-40 15-20 2-4	60-90	6-8	7-11	
4.	Chemical degreasing in the 2nd bath	Hot tub	NaOH KOH $\text{Na}_2\text{CO}_3$ $\text{Na}_3\text{PO}_4$ Liquid bottle	60-100 30-40 15-20 2-4	60-90	6-8	7-10	
5.	Washing	Bubble bath	Hot running water		60-90		0.5 -3	

6.	Washing	Bubble bath	Cold running water				0.5 -1	
7.	To feed		HCl	150-375	15-30		0.5 -3	Composition of AF-13 activating solution: 1. $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ 2. $\text{Na}_2\text{CO}_3$ 3. $\text{TiCl}_3$ 4. $\text{FeCl}_3$
8.	Washing	Bubble bath	Cold running water		15-30		0.5 -1	
9.	Washing	Bubble bath	Cold running water		15-30		0.5 -1	
10	Neutralizing soda solution	Bath	Calcined soda				0.5 -1	
11	Washing	Bubble bath	Warm running water		40-60		0.5 -1	
12	Processing in AF-13 activator solution with a mixture of compressed air	Bubble bath	AF-13 activating solution	0.5-1.0	30-40		1-3	
13	Washing	Bubble bath	Warm running water		40-60		0.5-1	
14	Phosphating	Hot tub	2nd composition of K P M – 2 concentrate	100	65-80		20	Acidity pH= 3.8-4.5
15	Washing	Bubble bath	Cold running water		15-30		0.5 -1	
16	Washing	Bubble bath	Hot running water		60-90		0.5-1	
17	Passivation	Hot tub	$\text{K}_2\text{Cr}_2\text{O}_7$ or $\text{Na}_2\text{Cr}_2\text{O}_7$	80-100	65-80			
18	Washing	Bubble bath	Warm running		40-60		0.5-1	

			water					
19	Washing	Bubble bath	Hot running water		60-90		0.5-1	
20	Washing	Bubble bath	Hot running water		60-90		0.5-1	
21	Compressed air drying						1-2	Compressed air must be dry
22	Drying				100-120		2-3	The details are dried in the drum processing
23	Disassembly of parts		Cloth gloves					
24	Quality control		Cloth gloves					
25	Paint	Paint brush						According to GOST
26	To soak up the details to give		Cloth gloves					

Electrode materials for water electrolyte lithium-ion batteries were reviewed. There are both cathodic and anodic oxides and polyanionic compounds.  $\text{LiMn}_2\text{O}_4$ ,  $\text{MnO}_2$ ,  $\text{LiCoO}_2$ ,  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ ,  $\text{LiFePO}_4$ ,  $\text{FePO}_4$ ,  $\text{LiMnPO}_4$  and others were considered as the cathode. The main problems of using these materials in the water system were the limited specific capacity and significant loss in the battery cycle. Experiments have shown that the limitation of the specific capacity is due to (1) the addition of  $\text{H}^+$  to the structure; (2) exchange during the  $\text{Li}^+/\text{H}^+$  cycle; (3) penetration of water into the structure and (4) dissolution of active materials in aqueous electrolytes. Many studies have addressed these issues by modifying the cathodes with additives or additives and by coating the material/electrolyte interface with all kinds of coatings or by changing the electrolyte composition.

$\text{VO}_2$  (B), spinel ( $\text{Li}_2\text{Mn}_2\text{O}_4$ ), layered  $\text{g-LiV}_3\text{O}_8$ , paramorph  $\text{VO}_2$ ,  $\text{V}_2\text{O}_5$ , Anatase  $\text{TiO}_2$  and polyanionic compounds  $\text{TiP}_2\text{O}_7$  and  $\text{LiTi}_2$  (Superionic current conductors of type  $\text{PO}_4$ )<sub>3</sub> were used. The main problems of these anode materials are the significant decrease in capacity during the electrochemical reaction and the limitation of practical potential compared to conventional lithium-ion batteries. Studies of anode materials have revealed the reasons for maintaining a low potential: (1) dissolution of active compounds; (2) irreversible structural changes, probably proton insertion, and (3) a spontaneous extraction reaction from lithium substituents leading to the formation of  $\text{LiOH}$  (aqueous solution) and hydrogen



are significantly improved by electrolytes with precisely controlled pH, type and concentration of lithium salt, residual  $\text{O}_2$ , and surface coating of active materials.

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