

EFFECT OF EXTRUSION MODIFIERS OF COATING MIXTURE ON ELECTRODE COATING THICKNESS DIFFERENCE*

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The method of oscillographing the signals generated by electromagnetic sensor, through the channel of which the electrodes pass at the outlet of the press coating head, was used to study the effect of extrusion modifiers of the coating mixture on thickness difference in manufacture of test electrodes UONI 13/55 of 4 mm diameter. The phenomenon of reduction of coating thickness difference under the effect of modifiers (CMC, alginates, cellulose or soda ash) is explained by that the dose of liquid glass added to the mixture gradually increases with the change of the type and increase of the content of modifier. Hydrating of modifying additive particles as a result of absorption of liquid glass moisture leads to thickening of the glass, and of its intergranular layers in the coating mixture, and to weakening of Hamaker interaction of filler grains. Probability of generation and accumulation of elastic stresses during extrusion application of the mixture on the rods becomes lower. The ability of the coating mixture flow to cover the rods in a uniform concentric layer at crimping of the electrodes becomes higher, accordingly. 6 Ref., 1 Table, 11 Figures.

Keywords: coated electrode manufacturing, coating mixture, coating thickness difference, extrusion modifiers, liquid glass, coating viscosity, electrode quality

Technological additives of natural origin or synthesized chemically are used in order to maintain a stationary pressure flow of coating mixtures during their extrusion deposition on the rods and ensuring a uniform concentric coating layer. They include silicates (kaolin, bentonite, muscovite mica), chemicals (soda, potash) or powders of organic hydrocolloids (cellulose, CMC, alginates). Plasticizing impact of the above modifiers is attributable to their densifying effect on the liquid glass binder in the coating mixture composition or suppression of the chemical reaction between the liquid glass and marble of the coating mixture. It is known that the silicates have structural moisture in their composition, and the base of alkali hydrocolloids consists of carbohydrates. It is important that their use does not lead to deterioration of metallurgical and other technological qualities of the manufactured electrodes.

The range of modifiers is quite broad, and the mechanism of their anticipated action began to be clarified only at the end of 1980s. This study provides analysis of its physico-chemical features and substantiation of rational methods of its realization.

Objects and procedure of investigation. Powders of organic hydrocolloids in the form of Na-CMC of 85CO grade from Namangan Chemical Plant and Na-alginate of Manutex RK/F grade of Alginate In-

dustries Ltd. (Great Britain), and of EC cellulose, as well as soda ash, were studied. Each of them was tested individually, just its content in the coating mixture was changed. Coating mixture not containing the above additives was studied for comparison. Investigation procedure described in detail in [1] was used.

Characteristics of grain composition of the charge were as follows: passing through 0063 mesh — 34.0 %, specific surface of 3700 cm²/g; grain packing density — 0.790.

NaK liquid glass with 3.05 module, $\rho = 1435 \text{ kg}\cdot\text{m}^{-3}$ and $\eta = 910 \text{ mPa}\cdot\text{s}$ was used in all the experiments, and the one with $\eta = 500 \text{ mPa}\cdot\text{s}$ was used in the experiment with the alginate. The coating mixture was prepared in single roller mixer.

The conditions of coating mixture preparation are given in the Table. The designations accepted in the Table are as follows:

- $t_{\text{cm}}, P_{\text{cr}}$ is the duration of preparation of coating mixture and pressure developed in the press head at electrode crimping;

- $e_i, e_{\text{sm}}, R_{\text{rms}}, S_r$ are the individual, sample mean, ranges and standard deviations of the values of coating thickness difference, calculated by the x_i and y_i coordinate components of coating thickness difference vector (CC CTDV) recorded on the oscillograms;

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Experimental conditions and results

Experiment number	Glass, coating mixture			Selective parameters of coating thickness difference, mm					Reject rate, %
	Dose, %	t_{cm} , min	P_{pr} , MPa	Charge number	N , pcs	e_{sm}	R_{rms}	S_r	
0 % CMC of 85CO grade in the charge									
1188	23.0	10.5	55.0 ¹	1.1	100	0.18	0.055	0.025	57.0
				1.2	100	0.23	0.050	0.015	61.0
				1.3	100	0.15	0.060	0.025	35.0
				2	90	0.16	0.060	0.030	26.0
0.5 % CMC of 85CO grade in the charge									
1205	24.4	7.0	52.0	1	100	0.07	0.060	0.025	0
				2	100	0.08	0.040	0.015	0
				3	100	0.09	0.035	0.015	0
1.0 % CMC of 85CO grade in the charge									
1306	27.0	5.0	52.0	1	100	0.11	0.035	0.025	2.0 ²
				2	80	0.14	0.025	0.010	0
				3	90	0.12	0.025	0.010	0
1.5 % CMC of 85CO grade in the charge									
1407	30.1	3.5	52.0	1	120	0.06	0.025	0.015	0
1.0 % alginate in the charge									
8848	28.7	7.0	53.0	1	100	0.03	0.035	–	0
				2	100	0.04	0.040	–	0
				3	80	0.02	0.025	–	0
2.0 % cellulose in the charge									
2313	28.8	8.0	44.0	1	65	0.04	0.030	0.015	0
				2	50	0.06	0.030	0.017	0
				3	90	0.04	0.043	0.056	0
2.5 % cellulose in the charge									
1814	28.8	8.0	48.0	1	70	0.01	0.009	0.004	0
				2	55	0.01	0.018	0.009	0
				3	45	0.02	0.023	0.017	0
1.2 % soda in the charge									
2408	31.6	4.0	40.0	1	100	0.04	0.040	0.015	0
				4	100	0.07	0.015	0.010	0
2.4 % soda in the charge									
2409	37.5	5.0	40.0	1	60	0.01	0.015	0.005	0
				5	60	0.03	0.023	0.010	0

Notes. ¹Crimping pressure at standard gap is not higher than 80 MPa. ²Starting portion of crimping was recorded on the oscillogram.

• $N = 100$ is the number of e_i measurements in the total population accumulated during 60 s. The oscilloscope tape speed is $10 \text{ mm} \cdot \text{s}^{-1}$, electrode crimping rate is 420 pcs per minute (2 measurements of x_i and y_i per three electrodes).

The e_{sm} , R_{rms} , S_r values were calculated by the moving averages technique, which is used to study the time series, when predicting, for instance the weather, currency rate, market prices, etc. With this purpose, similar to [1], the total data set was conditionally divided into 20 consecutive samples, each consisting from five e_i values following each other. Sample values of e_{sm} , R_{rms} , S_r , eliminating the noise, better reflect trends than e_i .

Investigation results. *Difference of thickness of coating from coating mixture without modifiers.* As was anticipated, unfavourable technological properties were found in the coating mixture without extrusion modifiers, designated by index 1188. Electrodes could

only be pressed from it, when the gap between the tip of the rod guide and the calibrating sleeve, i.e. press chamber volume, was doubled, compared to its standard size. Still, the mixture coats the rods in a nonuniform layer, and the crimping process essentially can not be adjusted. CC CTDV values in this experiment were registered continuously for 240 s. It follows from the data given in the Table that the produced electrodes had inadmissibly high and rather unstable values of coating thickness difference. The reject rate, i.e. the quantity of electrodes with above-norm values of coating thickness difference, is greater than 60 %.

The shape of oscillograms reveals that extrusion deposition of coating from mixture 1188 on the rods is a stochastic and multifactorial process. During crimping, both the horizontal and the vertical coordinate components of CTDV vary randomly. In this experiment, the amplitude and duration of x_i deviations from zero values is greater than that of y_i .

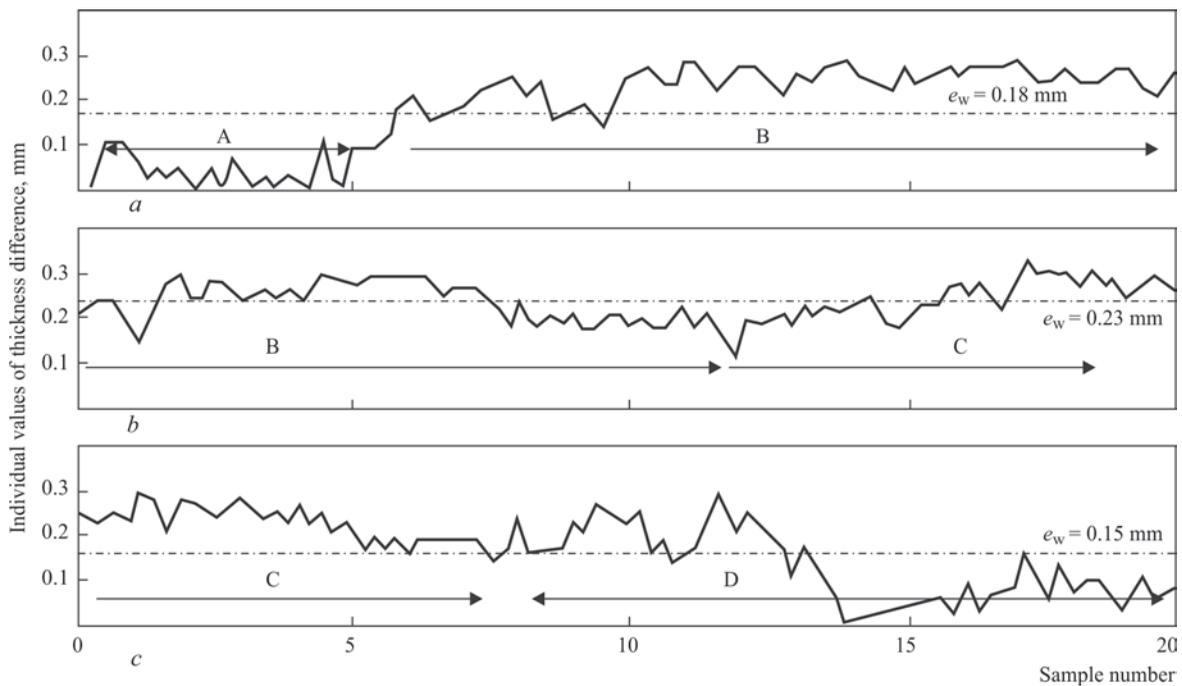


Figure 1. Evolution of e_i in electrodes pressed out of mixture 1188 (batch 1); *a-c* — designations of the starting (denoted by index 1.1 in the Table), medium (1.2) and end (1.3) portions of CTDV oscillograms

Judging by the orientation of stochastic deviations of x_i from x_0 from zero lines, they, most probably, are caused by the impact on the rod of the pressure of the column of coating mixture, injected from the cylinder into the press head. Mixture slipping on the rotating plate surface is obviously superposed on it.

The motive of periodical short-term and low-amplitude deviations prevails in the profile of CTDV vertical component. They can be caused by pulsating

passage of the mixture through vertically located divider windows in front of the press chamber.

Figure 1 shows the evolution of the calculated individual values of the difference of coating thickness of electrodes pressed from 1188 mixture. Arrows indicate the starting section of oscillogram A, sections of over-normative values of thickness difference B and C, as well as transition section D, within which the direction of evolution of thickness difference values changes. The wavelike elements of thickness difference evolution with a longer (A), and shorter (B) period and approximately the same amplitude are clearly visible.

One can see stochastic ripple with the frequency by orders of magnitude greater than that of the waves with periods A and B.

Figure 2 gives the shape of the trajectory of displacement of the projection of electrode core on the plane normal to the electrode axis. Indices 2N and 2K denote the initial and final branches of the phase diagram, and indices S and E show the beginning and end of each branch. A spiral broken in appearance is unwound between them, with the change of the right twist (solid line) into the opposite one (dash line), and simultaneous ripple of its longitudinal and transverse dimensional parameters. Initial branch designated by index 2N, pulsates along the direction of electrode displacement for approximately 30 s, remaining, mainly, within 0.20 mm, and the finish point of CTDV displacement almost coincided with the starting one. The final branch of the spiral, denoted by index 2F, contrarily reflects the low-amplitude ripple of CTDV

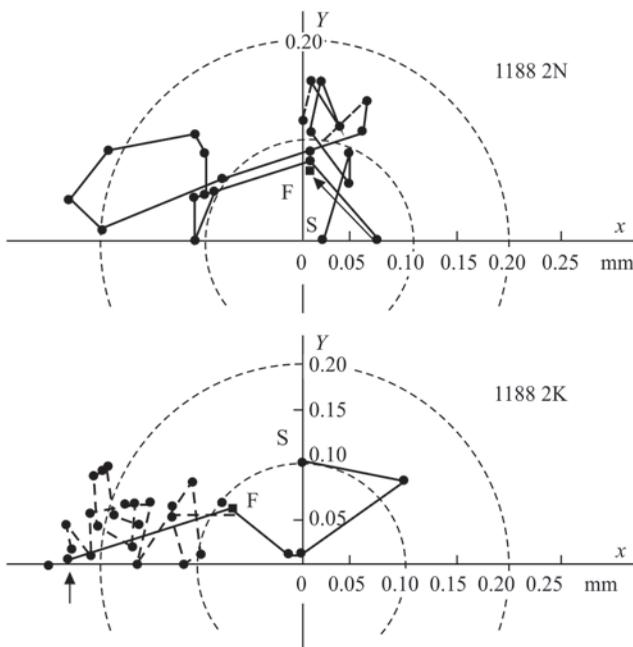


Figure 2. View of phase trajectories of CTDV obtained at crimping electrodes from batch 2 of mixture 1188 (for designations see the text)

tip, which during the same period of time, gradually shifting in the radial direction, finally goes beyond the circumference of admissible values of thickness difference of 0.20 mm diameter.

By the nature of the curves, given in Figures 1 and 2 it can be argued that during the crimping, which they reflect, it is impossible to regulate the process using the means available to the operator.

Impact of modifiers on coating thickness difference. At addition of organic hydrocolloids in the form of CMC (from 0.5 up to 1.5 %) and alginate (1 %) to the coating mixture, it becomes possible to produce electrodes from it with a standard gap in the press head, and mean sample values and characteristics of scatter of coating thickness difference decrease (see Table).

Coating mixtures with CMC. Starting from 0.5 % CMC, the profile of oscillograms of CC CTDV evolution, as well as the curves of e_i variation during crimping are smoothed the more, the greater is the proportion of CMC in the coating mixture (Figure 3).

Here, large-scale trends become stronger in e_{sm} , R_{rms} , S_r evolution, which are indicative of increase of electrode uniformity by e_i characteristic, and cases of over-normative thickness difference are not observed at all.

Naturally the histograms of e_i distribution and trajectories of CTDV phase diagrams are essentially densified, although the latter do preserve their spiral shape, not going beyond a circle of 0.15 mm diameter.

Coating mixtures with alginate. Judging by absolute values of e_{sm} , the technological properties of coating mixture with alginate are better than those of the mixture with the same CMC content. This is attributable to lower viscosity of the used liquid glass and its higher content, than in the coating mixture with index 1188, selected as the analog. By the characteristics of e_i value scatter, the electrodes from coating mixtures with the same content of alginate and CMC are identical.

Coating mixtures with soda. In terms of the ability to suppress the coating thickness difference, soda ash is a more effective extrusion modifier than CMC. In confirmation, it is sufficient to refer to Figure 4, from which it follows that in electrodes made from these coating mixtures, coating thickness difference does not exceed 0.10 mm. During one hour soaking, e_{sm} value somewhat rose. Therefore, soda particle swelling went on. Scattering of e_i value decreased, and none of the results exceeded 0.10 mm value.

Coating mixtures with cellulose. The data obtained at testing mixtures with 2.0 and 2.5 % cellulose are given in Figure 5. In electrodes from coating mixture designated by index 2313 (2 % cellulose), the scattering region of e_i values does not go beyond 0.10 mm, and for those pressed from coating mixture 1814 it

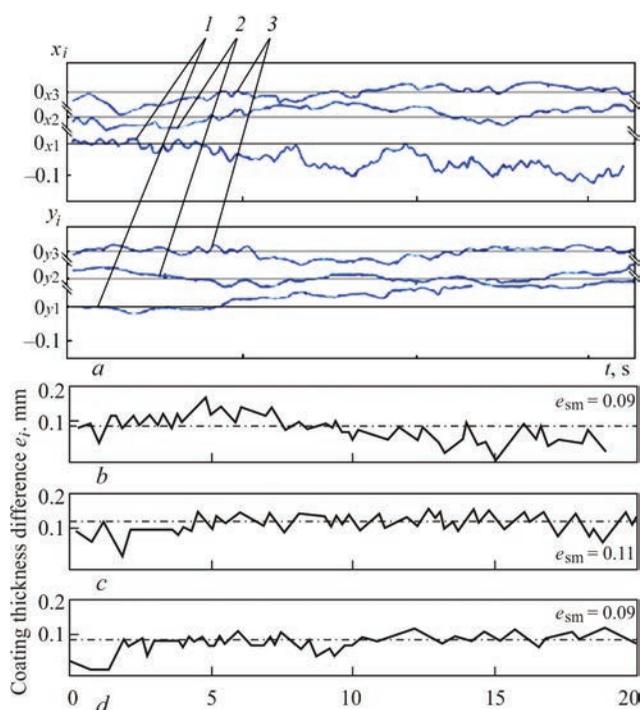


Figure 3. Copied portions of CC CTDV oscillograms (x_i and y_i) (a) and evolution of e_i during crimping of electrodes from mixtures 1205 (b), 1306 (c) and 1407 (d), containing 0.5 (1), 1.0 (2) and 1.55 (3) CMC, respectively

does not exceed 0.05 mm. Soaking of coating mixture 2313 changes the scattering profile of e_i , which, nonetheless, does not go beyond 0.10 mm. Soaking of coating mixture 1814 changes both the profile and the width of e_i scattering region. It, however, does not go beyond 0.05 mm even in the worst case.

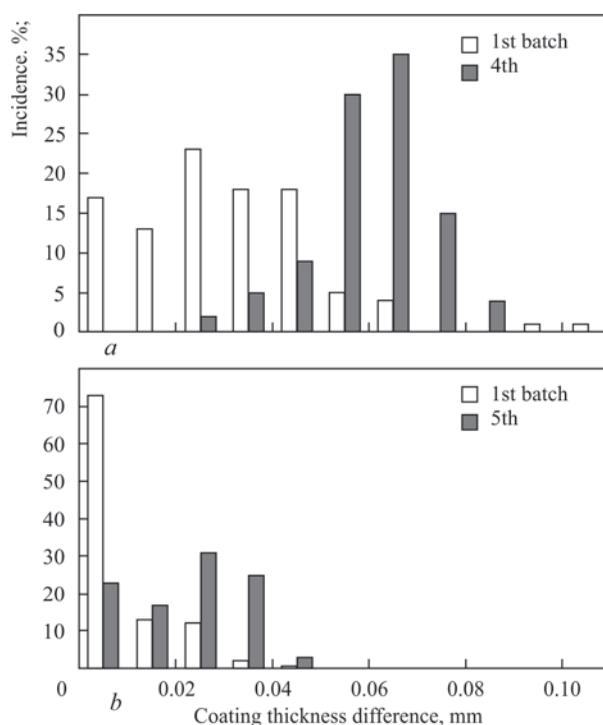


Figure 4. Density of distribution of e_i in electrodes made from coating mixtures 2008 (2 % soda, a) and 2009 (2.4 % soda, b)

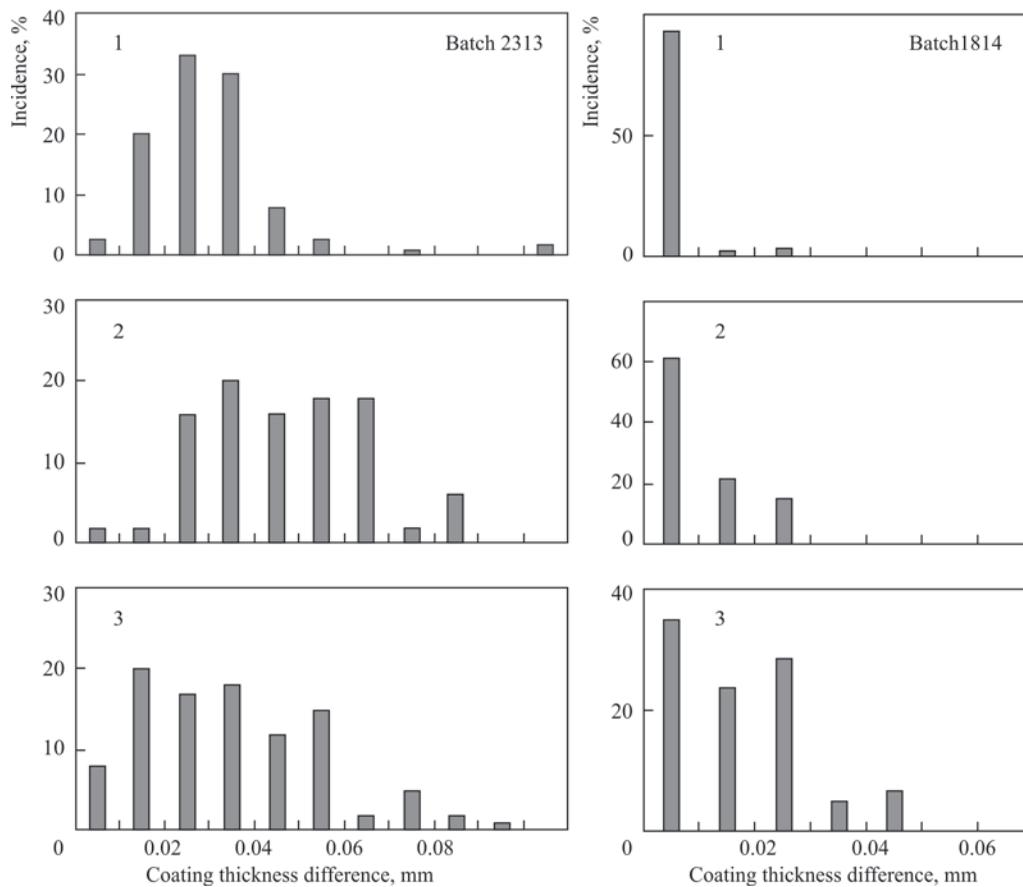


Figure 5. Density of e_i distribution in electrodes made from mixtures 2313 (2 % EC) and 1814 (2.5 % EC). 1–3 — batch number

Discussion. The shape of CC CTDV oscillograms, nature of variation of individual values of coating thickness difference, as well as the shape of CTDV trajectories, show that variation of the coating thickness difference during its extrusion application on the rods is a stochastic and multifactorial process.

The broken shape of curves $e_i = f(t)$ and chaotic displacement of the projections of CTDV trajectories to the plane, normal to electrode movement, bear the signs of fractality, inherent to the turbulent flow of liquids and Brownian motion of suspended particles, respectively. Randomly changing deviations from zero position of the horizontal, vertical, and in the case of low adaptability to fabrication of the coating mixture, of both the CC CTDV components can be regarded as the consequence of not only turbulent flow of the coating mixture, but also the complex power interaction of elastic (rod) and visco-elastic (enveloping coating mixture) elements of its flow in the press chamber. Elasticity is the cause, and viscosity is the means of damping of its relaxation process. The modulus of elasticity of the rod is constant. The results of our studies lead to the conclusion that the ratio of the modulus of elasticity and viscosity becomes smaller, and the probability of appearance of elastic turbulence of the mixture as the source of coating thickness difference in the pressure flow condition becomes high-

er as the coating mixture flow consistency becomes more rigid and its configuration becomes more complex, because of the change of its direction, degree, as well as the reduction rate.

The studied extrusion modifiers of coating mixtures are hydrophilic materials. CMC particles, dispersed in liquid glass, while sorbing moisture from it, swell and are wetted, by our data, up to 115 — 125 %; alginate particles — to a smaller degree and those of cellulose, apparently, to the smallest degree. Soda particles, while hydrating, can, by similar calculations, sorb up to 200 % of H_2O from liquid glass.

As a result of liquid glass dehydrating in the intergranular space, its viscosity and, accordingly, coating mixture extrusion resistance P_{ex} , as well as its plastic strength P_m , should increase. This is in agreement with the data given in Figure 6.

It follows from the Table that with the change of the kind and increase of modifier content, the dose of liquid glass added to the mixture increases as follows: from 23.0 % (mixture without plasticizer, designated by index 1188) up to 37.5 % (mixture with 2.45 % soda, index 2409). This is exactly how coating mixture thickening under the impact of the plasticizer is compensated. Here, the degree of the volume filling by charge grains is lowered, intergranular film of the binder becomes thicker, filler grain interaction be-

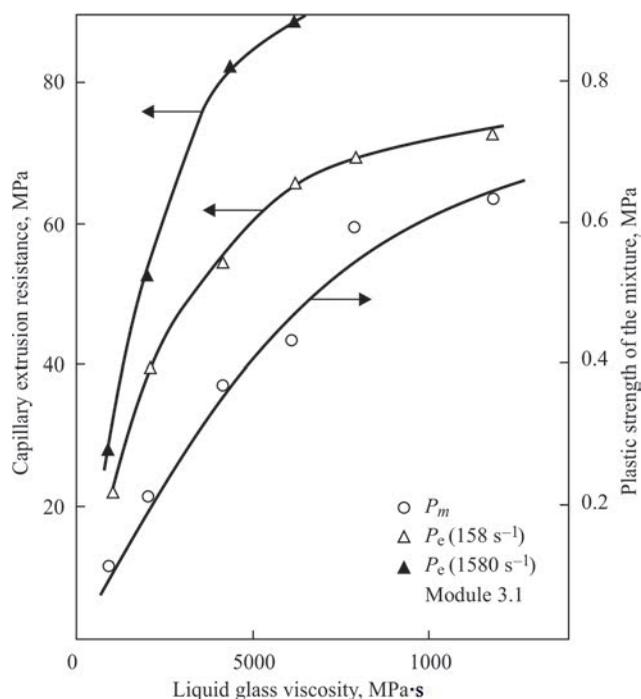


Figure 6. Variation of P_m and P_{ex} of coating mixture for UONI 13/55 electrodes, caused by increase of NaK liquid glass viscosity (25 wt.%)

comes weaker, that, eventually, should facilitate the activation nucleation of vacancies, required for shear displacement of grains of extruded mixture filler.

The probability of accumulation of elastic stresses in the coating mixture flow during its deposition on the rod is reduced. There is no need for their relaxation, for instance, by spontaneous transition of pressure flow of the mixture from a symmetrical one to eccentric circular cross-section, more favourable in energy terms [2].

It is important that weakening of the mixture consistency, due to increase of liquid glass content in it, just as the modifiers proper, did not become the cause for other defects of the coating and welds.

Production experience shows that the probability of appearance of coating thickness difference due to unfavourable extrusion characteristics of the mixture, as a rule, becomes lower, whereas its proneness to mechanical damage, because of its weak consistency, becomes higher. This is, in particular, indicated by the given in Figure 7 systematized and generalized by us annual results of acceptance testing of electrode products, performed by the State Acceptance Service of one of the electrode manufacturing enterprises.

Variation of the composition, characteristics and dose of liquid glass, on the one hand, and kind and concentration of the modifier, on the other, allows adjusting the mixture consistency, thus achieving favourable ratios of its extrusion properties (P_{ex} as a measure of energy intensity of the process of coating deposition by extrusion on the rods) and plastic

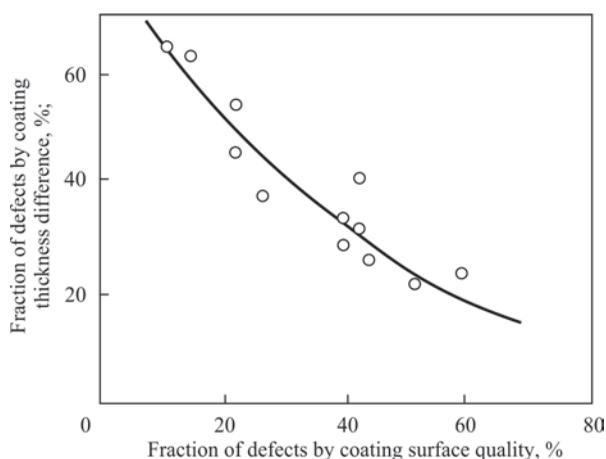


Figure 7. Interrelation of the fraction of electrodes rejected by thickness difference and surface quality of the coating (total reject rate of 3.5 %)

strength (P_m as a measure of raw coating resistance to mechanical damage). It follows from the data, given in Figure 8, that high-modulus liquid glasses, particularly those with a low viscosity, provide a larger range of P_m values of the mixture at the same variations of P_{ex} (hatched zone), than the low-modulus glasses. This is confirmed also by the given in the Table results of the experiment with electrode crimping from alginate-containing mixture.

Modifying effectiveness of the considered additives significantly depends not only on thickening ability, but also on their impact on the nature of physico-chemical interaction of the surface of filler grains with liquid glass. Polymer anions of asymmetrical molecules of CMC and alginates, dispersed in the intergranular film of the binder, as well as isomorphous

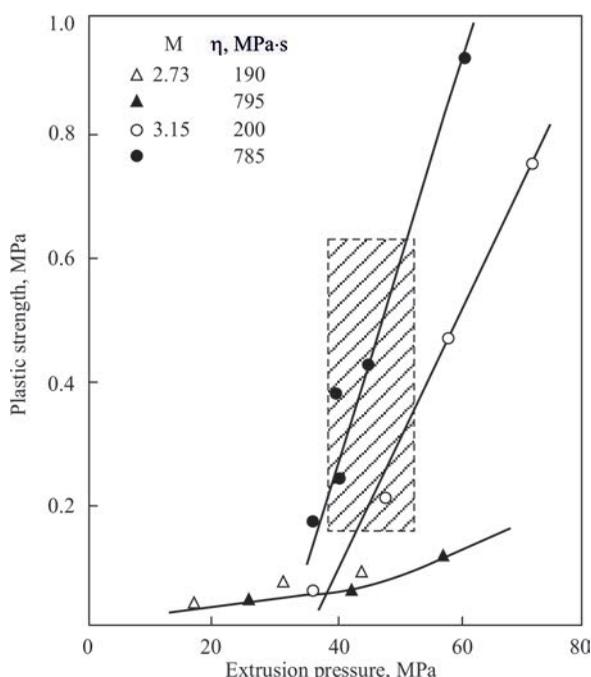


Figure 8. Impact of modulus and viscosity of KNa liquid glass on plastic strength of the coating mixture for ANO-27 electrodes

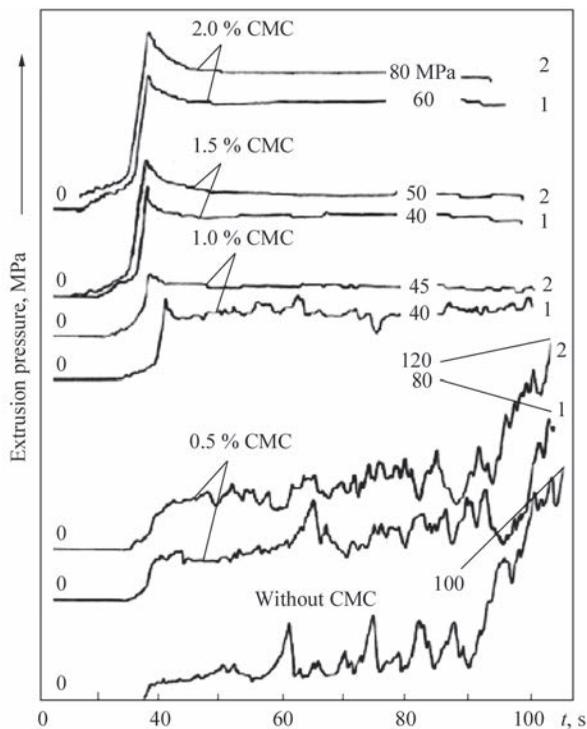


Figure 9. Curves of extrusion of coating mixture UONI 13/55 with different content of low-viscosity (1) and high-viscosity (2) CMC (NaK-liquid glass with 2.9 modulus and viscosity of 175 mPa·s, 22.5 wt.%)

carbonate anions, formed as a result of dissociation of soda molecules in it, are selectively sorbed by the hard surface of mixture filler particles. Similar to other surfactants, they block access of liquid-glass silicon-oxygen anions to it. This prevents the unpredictable thickening of the mixture, caused by increase of solvation barrier, and, therefore, hydrodynamic size of the grains, as well as ion-exchange transition into it of Ca from marble particles into the intergranular liquid-glass film.

The confrontation of alkali silicate and Na-CMC in an attempt to sorb on the surface of calcite grains is

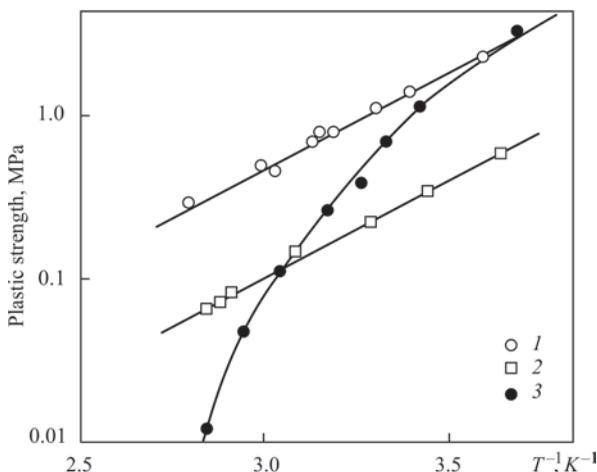


Figure 10. Temperature impact on softening of coating mixtures ANO-4 (1) and UONI 13/55 without soda (2) and with addition of 1.5 % soda (3)

confirmed by IR-spectroscopic and rheological studies. Results of IR-spectroscopic studies are considered by us in [3, 4]. Rheological results are given in Figure 9 in the form of extrusion curves, recorded by plastometer OB-1435 when studying UONI 13/55 mixtures, containing from 0 to 2 % of low-viscosity (68/350) and high-viscosity (68/920) Na-CMC. Capillaries of 4 mm diameter, and 40 mm length were used at average gradient of shear rate of 158 s^{-1} .

One can see that in mixtures without CMC and with 0.5 % CMC the nature of outflow is highly unstable — pressure pulsates and rises as the mixture is used, as a result of its dilatancy and liquid phase extraction. At such low concentrations the compared CMC compositions are incapable of blocking the processes of SOA sorption and calcium ion transition into liquid glass. This is in agreement with the given in the Table results of determination of coating thickness difference.

Flowing of the mixture containing 1 % of high-viscosity CMC, is stabilized and remains stable at concentrations, exceeding 1 %. Its low-viscosity modification is capable of ensuring stable extrusion only at 1.5 % and higher content.

At present several production variants of modifying the extrusion properties of mixtures are used, namely individually (CMC, alginate, soda and cellulose), CMC in combination with soda, as well as CMC in combination with cellulose.

In order to increase their technological effectiveness, it is recommended to use intensive mixers of counterflow type for mixture preparation. Up to 2 % water are added to the mixer, together with glass, slightly «dusting» the mixture with hydrocolloid powder before unloading from the mixer.

Kind, content and ratio of the proportions of extrusion modifiers of the mixture, in combination with the kind, module, viscosity and dose of liquid glass, optimum in terms of minimizing the coating thickness difference, are selected taking into account the features of the applied heat treatment and requirements to consumer properties of the electrodes.

So, for instance, it should be taken into account that at the beginning of electrode drying, soda increases the mixture proneness to thermal softening, which promotes formation of surface defects of the coating [5]. This is confirmed by shown in Figure 10 impact of temperature on P_m of ANO-4 and UONI 13/55 coating mixtures. The most intensive P_m lowering at heating was found exactly in the mixture containing soda, which was due to releasing (starting from $T = 40 \text{ }^\circ\text{C}$) of moisture absorbed by soda during the process of charge mixing with liquid glass. In mixtures containing organic hydrocolloids, the rate of thermal soft-

ening, contrarily, decreases, particularly, when their coarse modifications are used [4].

Felt tapes 3–5 mm thick have to be pasted on working surfaces of metal frames used for electrode transportation in continuous-operation drying furnaces, in order to prevent formation of dents or sticking of coatings, containing soda as extrusion modifier [6].

Moreover, soda is hygroscopic, and it lowers the hydrosorption resistance of the coating (Figure 11). This drawback cannot be removed by trivial technological measures.

Unlike soda, organic hydrocolloids in the coating are completely destructured in the standard mode of low-hydrogen electrode baking. Hygroscopicity of the coating, due to the formed pores and their ash residue, is much lower than that caused by soda, even in the most unfavourable case (see Figure 11). Nonetheless, it should be taken into account that hygroscopicity can increase, if the dose of glass required for compensation for mixture thickening by the modifier is exceeded too much.

Conclusion

Analysis of the results of the performed studies leads to the conclusion that powders of CMC, alginates, cellulose and soda ash really modify the coating mixture of low-hydrogen electrodes. Dispersed in the intergranular layer of liquid glass hydrophyllic particles of modifiers sorb moisture from it that results in increase of mixture viscosity. Increase of liquid glass dose is a means, «thinning» the mixture due to increasing the volume not taken up by the filler, and concentration of vacancies in it, which form during flowing.

While competing with silicon-oxygen anions (SOA) of liquid glass, the hydrated molecules of organic modifiers or soda carbonate ions of the same name drive SOA out of the interphase layer on the surface of grains of marble and other filler ingredients. The thus favourably transformed nature of the interphase layer, blocking of the transition of calcium ions into the liquid-glass film, reduction of the hydrodynamic particle size and EDL charge on filler grain surface, weaken the mixture consistency, as a result of

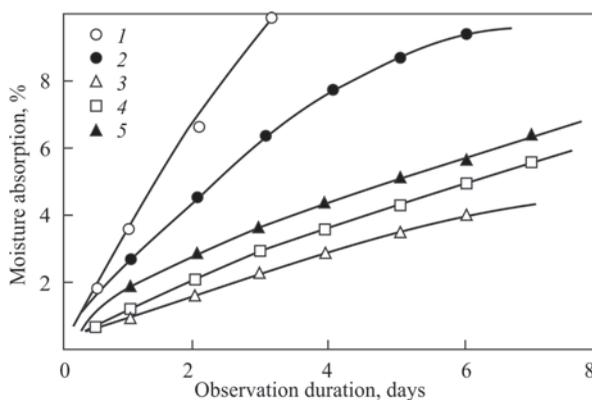


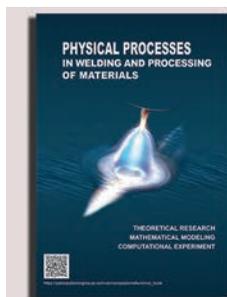
Figure 11. Comparison of hygroscopicity of coating of UONI 13/55 electrodes, made from mixture with 2.4 (1), 1.6 (2) soda, 1.5 (3), 1.0 (4) and 0 (5) % NaCMC

addition of liquid glass to it. Hydrated particles of the modifiers proper can be regarded as additional vacancies for mixture flow filler grains.

Combining certain kinds and concentrations of modifiers, on the one hand, with the needed characteristics and dose of liquid glass in the mixture, on the other, allows improving its consistency to the level, necessary for rheological support of the required quality of electrodes in terms of thickness difference, without detracting from their quality as regards other kinds of coating defects.

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