Some Properties of Electro-Deposited Fullerene Coatings

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ABSTRACT

This paper represents the results of investigations into thermal oxidation of electrodeposited fullerene-containing anodic coatings, the dependence of oxidation parameters on the composition of working solution. Some electrolysis products have been investigated by IR spectroscopic and X-ray phase methods.

The thermal parameters for oxidation of the coatings produced by electrolysis of TFE solutions with additives and without them are given. The dependence of the chemical composition of the coatings on the type of additive has been established. It has been shown that in heating the coating, deposited electrically from the TFE solution, fullerenes are sublimated and graphitized.

IR transmission spectra of electrolysis products, produced from the TFE solutions with KBr or KOH additives and without them, and also diffractograms of coatings, deposited electrically from the TFE solution without additives and with KOH, are represented. Analysis of IR transmission spectra and diffractograms confirms heterogeneity of the chemical composition of the coatings, deposited electrically from the TFE solution with KBr or KOH additives. This solution is a mixture of the fcc lattice fullerenes and fullerene-containing compounds.

Keywords: fullerene solution, ethanol, toluene, anodic coating, oxidation, electrolysis

1 INTRODUCTION

Earlier we have demonstrated the principal possibility to produce fullerene-containing coatings on metal electrodes by the electrochemical method and the dependence of their structure on the chemical composition of the working solution and experimental conditions [1].

This work represents the results of investigations into thermal oxidation of electro-deposited fullerene-containing anodic coatings, the dependence of oxidation parameters on the composition of the working solution. Some electrolysis products have been investigated by IR spectroscopic and X-ray phase methods. Based on comparison of the obtained results, we have made conclusions about phase and chemical compositions of the substances investigated.

2 EXPERIMENTAL CONDITIONS

The anodic coatings on Ni-electrodes have been investigated. The coatings have been produced by the electrochemical method from the fullerenes solution in toluene (TF). The base electrolyte for this solution was either ethanol (TFE solution), or ethanol with one of the additives of KOH, KBr, $LiClO_4$ type, or KCl (TFE with additives). The difference in potentials of electrodes depended on the composition of the working solution and corresponded 600-1600 V for the TFE solution and 10-80 V for the TFE solution with above additives.

Thermal-gravimetric studies have been performed on the derivatograph Q-1500D in conditions of dynamic heating the coatings in air at 20-1000^{o}C. IR transmission spectra of electrolysis products, which were mechanically separated from the electrodes (powders), have been registered using the two-beam spectrophotometer Specord 75-IR in the range of wave numbers 400 to 4000 cm^{-1}.

3 RESULTS AND DISCUSSION

Oxidation of the coatings starts at T≥200^{o}C. The temperature range and the temperature of the maximum rate of oxidation depend on the composition of the working solution. In this case the maximum rate of oxidation is observed at T_{max}=435^{o}C. Oxidation of the coating produced from the TFE solution with KOH additive is characterized by the appearance the shoulder at T_1=350^{o}C and the sharp peak at T_{max}=470^{o}C in the DTG curve (Fig.1, curve 2).

The observed peculiarities in oxidation of anodic coatings produced from the TFE solutions with additives and without them indicate the difference in their chemical composition. According to the literature data [2, 3], the temperature of the maximum rate of fullerenes oxidation is in the temperature range 420 to 570^{o}C and depends on dispersivity and the degree of fullerenes crystallinity.

Comparison of the experimental thermal-gravimetric parameters with the literature data on fullerenes oxidation allows us to suppose that the anodic coating produced from the TFE solution without additives comprises fullerenes with the oxidation temperature ≈435^{o}C. This is also confirmed by IR spectroscopic and X-ray phase studies of the electrolysis products.

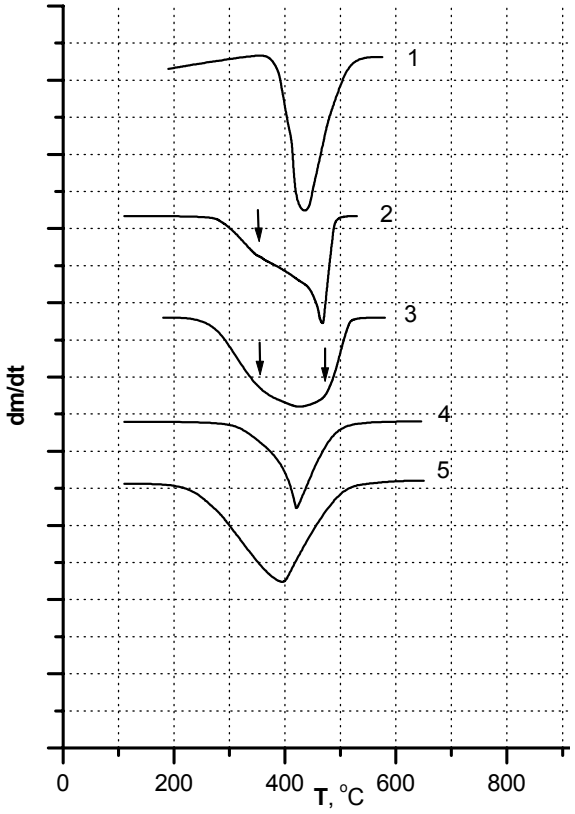

Figure 1: DTG curves for oxidation of anodic coatings on Ni-electrodes. The coatings have been produced by electrolysis of TFE solutions with additives and without them: 1 – without additives; 2 – KOH; 3 – KBr; 4 – $LiClO_4$; 5 – KCl.

Table 1: Parameters for oxidation of anodic products after electrolysis of TFE solutions with additives and without them.

#	Composition of electrolyte	Beginning interaction T,°C	DTG		
			T_{max}, °C	T_1, °C	T_2, °C
1	TFE	200	435		
2	TFE + KOH	280	470	350	
3	TFE + KBr	240	419	350	470
4	TFE + $LiClO_4$	280	419		
5	TFE + KCl	200	395		

4 CONCLUSIONS

1. The thermal parameters for oxidation of the coatings produced by electrolysis of TFE solutions with additives and without them are given. The dependence of the chemical composition of the coatings on the type of additive has been established.
2. It has been shown that in heating the coating that was deposited electrically from the TFE solution fullerenes are sublimated and graphitized.
3. IR transmission spectra of the electrolysis products that were produced from the TFE solutions with KBr or KOH additives and without them, and also diffractograms of the coatings that were deposited electrically from the TFE solution without additives and with KOH are represented. Analysis of IR transmission spectra and diffractograms confirms heterogeneity of the chemical composition of the coatings that were deposited electrically from the TFE solution with KBr or KOH additives. This solution is a mixture of fullerenes of the fcc lattice and fullerene-containing compounds. The coating that was deposited electrically from the TFE solution without additive comprises fullerenes of the fcc lattice and is a mono-phase system.

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