DERIVATOGRAFIC INVESTIGATION INTO THERMAL STABILITY OF DEPOSITES FORMED IN ELECTRIC ARC SPUTTERING OF ME_1-ME_2-C COMPOSITES

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One of the products in electric arc synthesis of fullerenes is the deposit that mainly comprises multi-wall carbon nanotubes and some amount of graphitized carbon. Metal additives introduced into the deposits are known [1, 2, 3] to affect significantly physical, chemical and other properties of the product. This report represents results of the investigation into the effect of Zr-Fe, Zr-Co, Zr-Cu, Zr-Ni, Zr-Ti, Zr-Mn, Zr-Ti-Mn, Hf-Fe, Hf-Ni, Co-Mg, Co-Ni, Co-Ti, Co-W, Ni-Fe-Cr, Al+Mm+Fe, Si alloys that are introduced into the composition of a graphite anode on oxidation of formed deposites.

Thermal gravimetric studies and differential thermal analysis of deposits have been performed using the derivatograph Q-1500D in conditions of dynamic heating in air from room temperature to 1000 °C. The obtained results are given in Fig.1-3 and in Table.

It has been established that considerable oxidation of deposits starts over 400 °C. Deposits with Zr-Fe, Co-Ni, Ni-Fe-Cr, Co-Ti additives are oxidized in the temperature range of 425-770 °C (Fig.1a-c, table). Rather wide peaks with temperature maximums in the range of 615-678 °C in DTG and DTA curves are characteristic of this process excluding the deposit with Zr-Fe. Two peaks (T_{max}= 580, 655 °C) are observed in the DTG curve for oxidation of the deposit with Zr-Fe alloy.

Table. Results of thermal studies of deposits with different catalysts.

№	Alloys	DTG					DTA		Temperature range for decomposition, °C
		$T_{1\,max}$, °C	$T_{2\,max}$, °C	$T_{3\,max}$, °C	$T_{4\,max}$, °C	$T_{5\,max}$, °C	$T_{1\,max}$, °C	$T_{2\,max}$, °C	
1	Zr-Fe	580	655				585	675	465-750
2	Zr-Co		665	715			650		450-795
3	Zr-Cu	615		710			600	680	430-775
4	Zr-Ni				748		725		515-880
5	Zr-Ti				761	785	750		525-965
6	Zr-Mn		678		725		720		465-815
7	Zr-Ti-Mn	620	678		742	777	640	735	430-940
8	Hf-Fe			711	728		675		490-830
9	Hf-Ni		650		735	770	645	730	400-855
10	Co-Mg		655		735		660	723	400-800
11	Co-Ni	615					598		445-700
12	Co-Ti		678				677		500-770
13	Co-W			709			700		460-860
14	Ni-Fe-Cr	626					640		425-765
15	Al+Mm+Fe	625		715			723		400-830
16	Si			710	728	776	760		525-845

In the DTA curve two overlapped exopeaks (T_{max}=585, 675 °C) correspond the above peaks.

The temperature range for oxidation of deposits with alloys that are shown in Fig.2a-c is wider (400-940 °C). DTG and DTA curves for oxidation of these deposits exhibit more complex behavior and have not less than two peaks both in lower (T_{max}=615-678 °C) and higher temperature (T_{max}=710-777 °C) ranges.

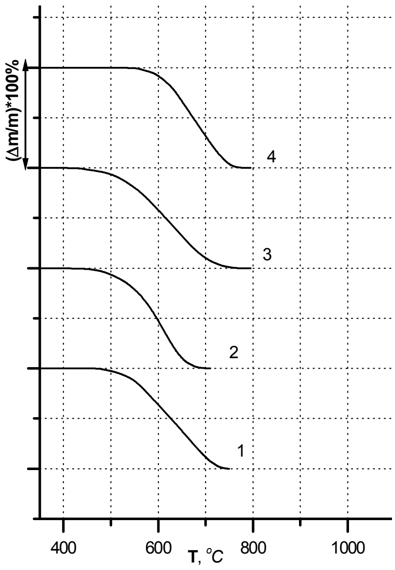

Fig.1a. TG curves for oxidation of deposits with alloys: Zr-Fe (1), Co-Ni (2), Ni-Fe-Cr (3), Co-Ti (4)

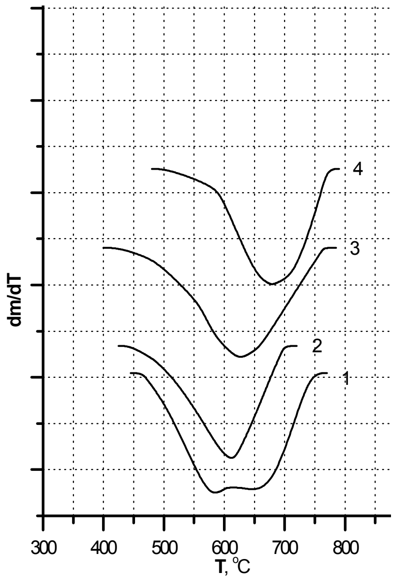

Fig.1b. DTG curves for oxidation of deposits with alloys: Zr-Fe (1), Co-Ni (2), Ni-Fe-Cr (3), Co-Ti (4)

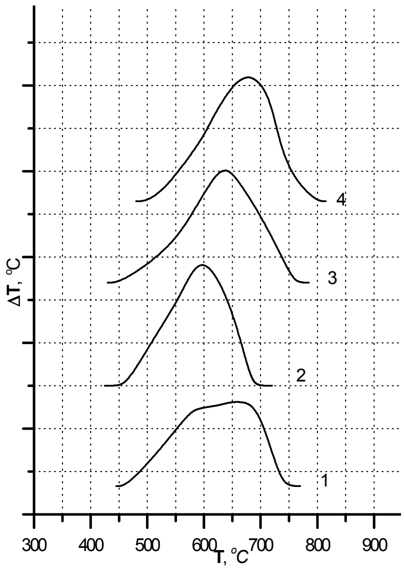

Рис. 1в DTA-кривые окисления депозитов со сплавами: Zr-Fe (1), Co-Ni (2), Ni-Fe-Cr (3), Co-Ti (4)

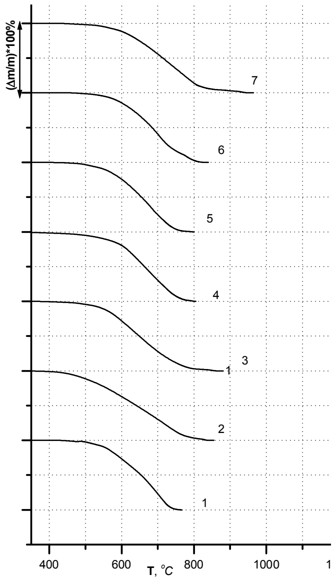

Fig.2a. TG curves for oxidation of deposits with alloys: Zr-Cu (1), Al+Mm+Fe (2), Hf-Ni (3), Co-Mg (4), Zr-Co (5), Zr-Mn (6), Zr-Ti-Mn (7)

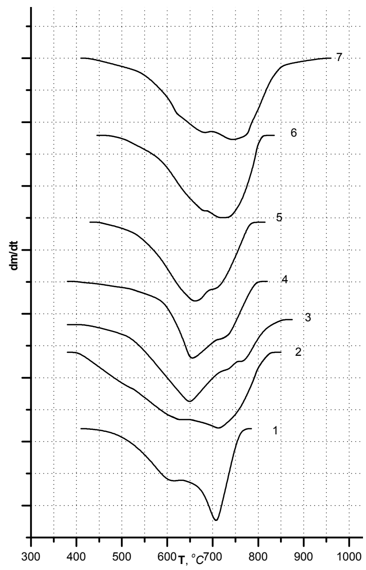

Fig.2b. DTG curves for oxidation of deposits with alloys: Zr-Cu (1), Al+Mm+Fe (2), Hf-Ni (3), Co-Mg (4), Zr-Co (5), Zr-Mn (6), Zr-Ti-Mn (7)

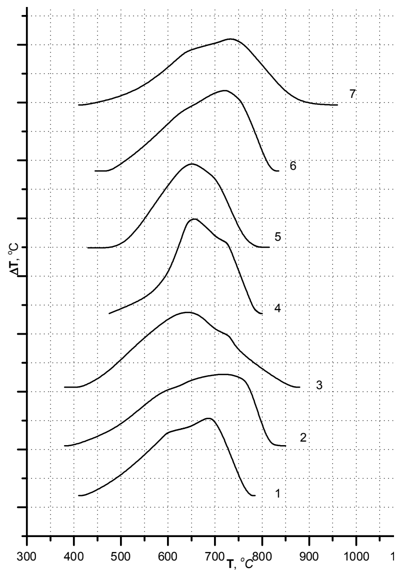

Fig.2c. DTA curves for oxidation of deposits with alloys: Zr-Cu (1), Al+Mm+Fe (2), Hf-Ni (3), Co-Mg (4), Zr-Co (5), Zr-Mn (6), Zr-Ti-Mn (7)

The main feature of high-temperature behavior of deposits with Co-W, Si, Hf-Fe, Zr-Ni, Zr-Ti alloys (Fig.3a-c) is that the maximum rates of their oxidation are observed above 700 °C. For these deposits, from one (Co-W, Zr-Ni alloys) to 2-3 peaks (Si, Hf-Fe, Zr-Ti alloys) are observed in the narrow temperature range in the DTG curves. In the last case there appears a number of overlapped exopeaks in the DTA curves (Fig.3c).

Hence the performed investigations have shown that temperature ranges, number and forms of peaks in DTG and DTA curves, temperatures for the maximum rate of deposit oxidation varies depending on the chemical composition of introduced additives. Literature data [2, 3] have shown that introduction of additives of different metals in little amounts into the composition of an anode affects considerably the structure and the

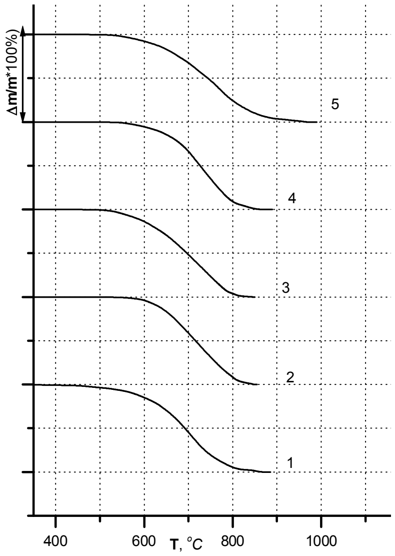

Fig.3a. TG curves for oxidation of deposits with alloys: Co-W (1), Si (2), Hf-Fe (3), Zr-Ni (4), Zr-Ti (5)

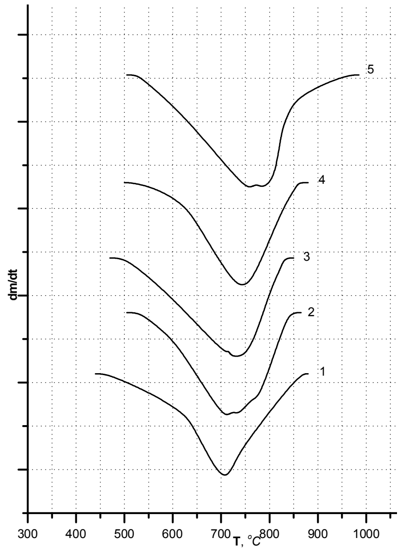

Fig.3b. DTG curves for oxidation of deposits with alloys: Co-W (1), Si (2), Hf-Fe (3), Zr-Ni (4), Zr-Ti (5)

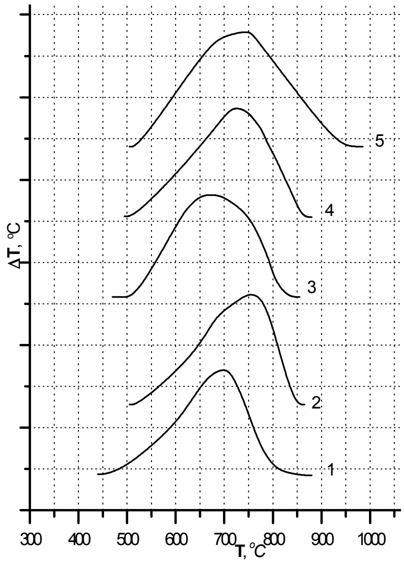

Fig.3c. DTA curves for oxidation of deposits with alloys: Co-W (1), Si (2), Hf-Fe (3), Zr-Ni (4), Zr-Ti (5)

configuration of nanotubes. X-ray structure studies have shown [5, 6] that besides the main graphite phase the above deposits comprise metals, carbides and intermetallic compounds. Introduction of metal into the space among nanotubes and inside the nanotubes gives rise to the change of the chemical composition and the electron state, and creates distortion in the structure of multilayer nanotubes what has a considerable effect on their thermal stability.

Conclusions

The performed investigations have shown that introduction of different additives into the anode composition changes considerably thermal stability of deposits.

References

1. Елецкий А.В. Эндоэдральные структуры. Успехи физических наук, 2000, 170 (2), с.113-142.
2. Раков Э.Г. Методы получения углеродных нанотрубок. Успехи химии, 2000, 69 (1), с. 41-59.
3. Елецкий А.В. Сорбционные свойства углеродных наноструктур. Успехи физических наук, 2000, 174 (11), с. 1191-1231.
4. Рогозинская А.А., Клочков Л.А., Шапошникова Т.И. и др. Рентгеноструктурное исследование депозитов, сформировавшихся при электродуговом распылении композитов Me_1-Me_2-C. Водородное материаловедение и химия углеродных материалов. ICHMS'2003. Киев, 2003, с. 652-653.
5. Шульга Ю.М., Щур Д.В., Мухачев А.П. и др. Исследование катодных депозитов, образующихся при электродуговом распылении Zr-M-графитовых электродов. Водородное материаловедение и химия углеродных материалов. ICHMS'2003. Киев, 2003, с. 452-453.