X-RAY INVESTIGATION OF DEPOSITS FORMED BY ELECTRIC ARC SPUTTERING COMPOSITES Me_1-Me_2-C

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Introduction

In connection with the perspective of using nanostructural carbon in different fields of industry and techniques, the materials based on it attract heightened interest. Electronics gives a particular place to these materials due to their physical-chemical properties.

One of the products formed after the electric arc synthesis of fullerenes is deposit-a growth forming on the cathode. Deposit mainly consists of multi-wall carbon nanotubes and some quantity of graphitized mass of carbon.

As deposit is formed at high temperatures and under influence of power electric field, it in some or other form is of interest as the raw material for electronic industry.

Additions introduced into the deposit by intercalation, encapsulation and other methods have a strong influence on physical-chemical, electric and other properties of the product what permits the extension of their application.

Results and discussion

In the work presented we have made X-Ray investigation of the products produced by electric arc evaporation of composites M_1+M_2+C (where M_1 and M_2 are different transition metals)

We have used the following composites: (Hf+Ni+C); (Hf+Fe+C); (Co+Mg+C); (Co+Ti+C); (Co+W+C); (Co+Ni+C); (Fe+Ni+C); (Fe+Ni+Cr+C); (Si+C):

Previously the deposits were subjected to grinding during 3 hours in the planetary mill. X-Ray photography of the ground deposits was made on X-Ray apparatus DRON-3M in filter CuKα-radiation followed by interpretation of the diffractograms. The position of the diffraction maximums was determined according to the center of gravity of lines.

X-Ray investigation of the deposit produced from the graphite anode without addition of metal catalysts showed that besides multi-layer carbon nanotubes the deposit consisted of two phase-hexagonal graphite and rhombohedral graphite with slightly increased interplane distances calculated by the center of gravity of the diffraction line (Fig.1).

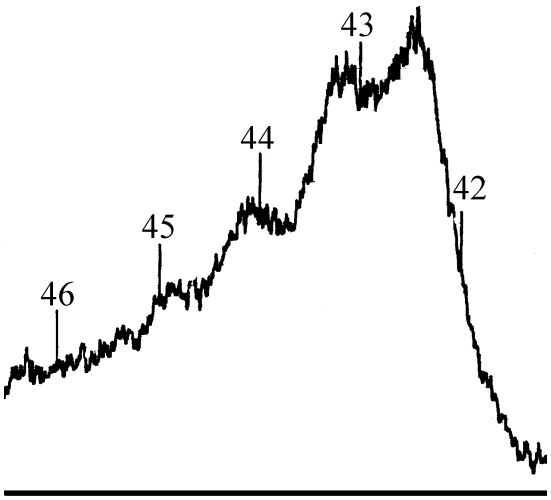

Fig. 1

After adding the metal catalysts the main phase in deposits remains graphite in two modifications (hexagonal phase predominates). Depending on the structure of the compositional addition, there appear carbides and intermetallides as well (Table 1).

So, after adding Si into the graphite row mix there appears silicon carbide β SiC (cubic modification with α=4,358Å) and traces of α-SiC (hexagonal modification).

The composition (Hf+Ni) added into the graphite raw mix results in the formation of hafnium carbide HfC of the stoichiometric composition with the lattice constant α=4,636 Å and very small amount of Ni_3C (Fig. 2).

The composition (Nf +Fe) forms in the deposit the HfC phase of the stoichiometric composition and very small amount of solid solution Hf(Fe)C. The substitution of hafnium atoms for iron ones leads to the decreases in the lattice constant of hafnium carbide (Table 1)

Co+Mg added into the graphite row mix of the anode results in the formation on the cathode deposit magnesium carbide Mg_2C_3 with the hexagonal lattice and very small amount of Co_2C.

Composite (Co+Ti) forms in the deposit titanium carbide with the stoichiometric composition.

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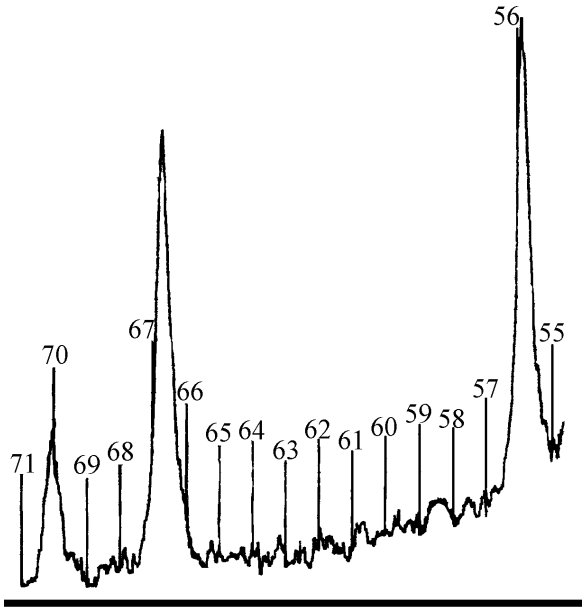

Fig. 2

Composite (Co+W) added creates in the deposit WC cubic and α-WC hexagonal phases in insignificant amount.

(Co+Ni) added into the anode raw mix forms in the deposit continuous solid solution Ni (Co). The small increase in the nickel lattice constant indicates it. This increase is due to the larger Co atoms in comparison with NICKEL atoms (Table 1)

When composition (Fe-Ni) used, in the deposit solid solution γ-Fe (Ni) forms where Ni stabilizes the γ-Fe lattice. In deposit, after electric arc evaporation of the anode with addition of (Fe-Ni-Cr), we have observed γ-Fe (Ni) and Cr_3C_2 phases (Table 1).

Table 1

Catalyst	Phase structure of deposit	*a*, Å	V, Å^3	**R**ат Me, Å
Si	C (graphite); βSiC α-SiC	4,358	82,768	Si 1,17к
Hf+Ni	C (graphite); HfC stoich. Ni_3C traces	4,636	99,639	Hf 1,59
Hf+Fe	C (graphite); HfC stoich. Hf(Fe)C	4,636 4,605	99,639 97,653	Hf 1,59 Fe 1,27
Co+Mg	C (graphite); Mg_2C_3 Co_2C	–	–	Mg 1,60 Co 1,26
Co+Ti	C (graphite); TiC stoich. CoTi	4,324	80,846	Ti 1,45 Co 1,26
Co+W	C(graphite); WC cub. α-Wc hex.	4,220	75,151	W 1,40
Co+Ni	C(graphite); Ni(Co) s.sol.	3,529 3,524Ni ref.	43,950	Ni 1,24 Co 1,26
Fe+Ni	C(graphite); γ-Fe(Ni) s.sol.	γ-Fe(Ni) 3,595 γ-Fe 3,645 ref.	46,462 48,427	Fe 1,27 Ni 1,24
Fe+Ni+Cr	C(graphite); γ-Fe(Ni) sol. sol. Cr_3C_2	γ-Fe(Ni) 3,598 γ-Fe 3,645 ref.	46,578 48,427	Cr 1,28 Fe 1,27 Ni 1,24

Conclusion

In the course of the work it has been established that the additions of transition metals in the evaporated electrodes during electric arc synthesis are presented in deposits as simple or complex carbides. Individual physical-chemical properties of each composition depend on the nature of the metal.

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РЕНТГЕНОСТРУКТУРНОЕ ИССЛЕДОВАНИЕ ДЕПОЗИТОВ, СФОРМИРОВАВШИХСЯ ПРИ ЭЛЕКТРОДУГОВОМ РАСПЫЛЕНИИ КОМПОЗИТОВ Me_1-Me_2-C

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Введение

В связи с перспективой использования наноструктурного углерода в различных областях производства и техники материалы на его основе привлекают в последнее время к себе повышенное внимание. Особое место этим материалам отводится в электронике благодаря присущим им физико–химическим свойствам.

Одним из продуктов электродугового синтеза фуллеренов является депозит – нарост, образующийся на катоде. Депозит в основном состоит из многостенных углеродных нанотрубок и некоторого количества графитизированной массы углерода.

Поскольку депозит формируется при высоких температурах и под воздействием мощного электрического поля, он в той или иной форме представляет интерес как сырье для электронной промышленности. Добавки, вводимые в депозит интеркалированием, инкапсулированием и другими методами, сильно влияют на физико-химические, электрические и другие свойства продукта, что позволяет расширить область их применения.

Обсуждение результатов

В настоящей работе проведено рентгеноструктурное исследование продуктов электродугового испарения композитов M_1+M_2+C (где M_1 и M_2 - различные переходные металлы).

Использовали следующие композиты: (Hf+Ni+C); (Hf+Fe+C); (Co+Mg+C); (Co+Ti+C); (Co+W+C); (Co+Ni+C); (Fe+Ni+C); (Fe+Ni+Cr+C); (Si+C).

Предварительно депозиты подвергались размолу в течение 3 часов в планетарной мельнице. Рентгеновская съемка размолотых депозитов производилась на рентгеновском аппарате ДРОН-3М в фильтрованном CuKα-излучении с последующей расшифровкой дифрактограмм. Положение дифракционных максимумов определялось по центру тяжести линий.

Рентгеноструктурное исследование депозита, полученного из графитового анода без добавок металлических катализаторов, показало, что, кроме многослойных углеродных нанотрубок, депозит состоит из двух фаз – графита гексагонального и графита ромбоэдрического со слегка увеличенными межплоскостными расстояниями, рассчитанными по центру тяжести дифракционной линии (рис. 1).

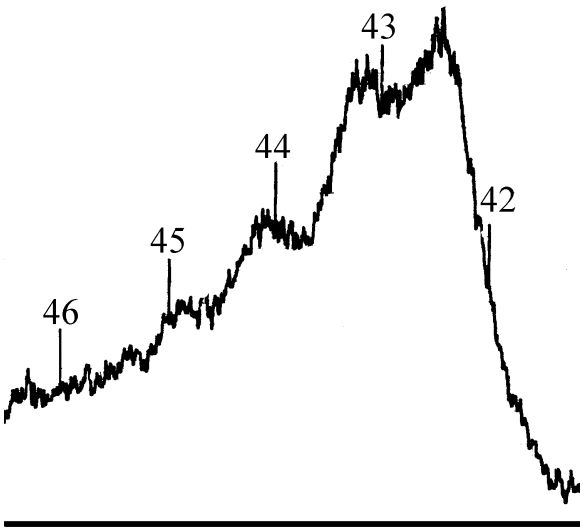

Рис. 1.

После добавления металлических катализаторов основной фазой в депозитах остается графит в двух модификациях (с преобладанием гексагональной фазы). Появляются также карбиды и интерметаллиды в зависимости от состава композиционной добавки (табл. 1).

Так, после добавления Si в графитовую шихту в депозите появляются карбиды кремния β – SiC (кубическая модификация a=4,358Å) и следы α-SiC (гексагональной модификации).

Добавление композиции (Hf+Ni) в графитовую шихту приводит к образованию карбида гафния HfC стехиометрического состава с периодом решетки a=4,636Å и очень малого количества Ni_3C (рис. 2).

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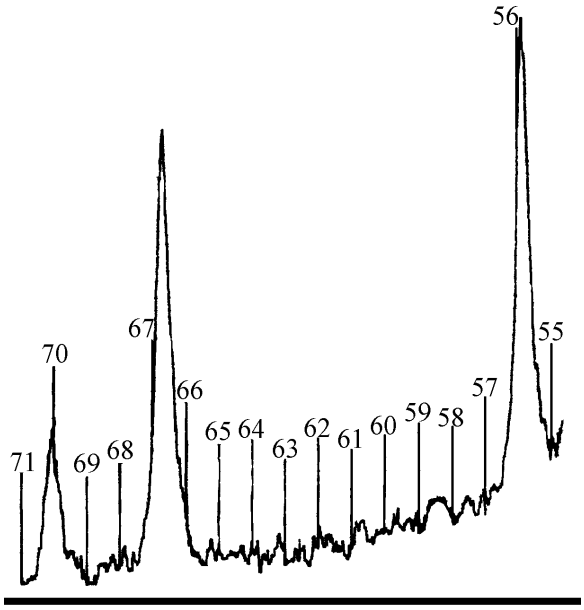

Рис. 2.

Композиция (Hf+Fe) образует в депозите фазы HfC стехиометрического состава и в очень малом количестве твердый раствор Hf(Fe)C. Замещение атомов гафния атомами железа приводит к уменьшению периода кристаллической решетки карбида гафния (табл. 1).

Добавка (Co+Mg) в графитовую шихту анода приводит к образованию в катодном депозите карбида магния Mg_2C_3 с гексагональной решеткой и очень малого количества Co_2C.

Композит (Co+Ti) образует в депозите карбид титана стехиометрического состава.

Добавка композита (Co+W) создает в депозите фазы WC кубический и α-WC гексагональный в незначительном количестве.

При добавке (Co+Ni) в шихту анода в депозите образуется непрерывный твердый раствор Ni(Co), о чем свидетельствует небольшое увеличение периода решетки никеля, связанное с более крупными размерами атомов Co по сравнению с атомами никеля (табл. 1).

При композиции (Fe-Ni) в депозите образуется твердый раствор γ-Fe(Ni), где Ni стабилизирует решетку γ-Fe. В депозите после электродугового испарения анода с добавкой (Fe-Ni-Cr) наблюдаем фазы γ-Fe(Ni) и Cr_3C_2 (табл. 1).

Таблица. 1

Катализатор	Фазовый состав депозита	*a*, Å	V, Å^3	$\mathbf{R}_{ат.}$ Me, Å
Si	C (графит); βSiC α-SiC	4,358	82,768	Si 1,17к
Hf+Ni	C (графит); HfC стех Ni_3C следы	4,636	99,639	Hf 1,59
Hf+Fe	C (графит); HfC стех Hf(Fe)C	4,636 4,605	99,639 97,653	Hf 1,59 Fe 1,27
Co+Mg	C (графит); Mg_2C_3 Co_2C	–	–	Mg 1,60 Co 1,26
Co+Ti	C (графит); TiC стех CoTi	4,324	80,846	Ti 1,45 Co 1,26
Co+W	C(графит); WC куб α-Wc гекс.	4,220	75,151	W 1,40
Co+Ni	C(графит); Ni(Co)тв. р-р	3,529 3,524Ni лит.	43,950	Ni 1,24 Co 1,26
Fe+Ni	C(графит); γ-Fe(Ni) тв. р-р.	γ-Fe(Ni) 3,595 γ-Fe 3,645 лит.	46,462 48,427	Fe 1,27 Ni 1,24
Fe+Ni+Cr	C(графит); γ-Fe(Ni) тв. р-р. Cr_3C_2	γ-Fe(Ni) 3,598 γ-Fe 3,645 лит.	46,578 48,427	Cr 1,28 Fe 1,27 Ni 1,24

Выводы

В ходе работы было установлено, что добавки переходных металлов в испаряемые электроды при дуговом синтезе присутствуют в депозитах в виде простых или сложных карбидов. Индивидуальные физико-химические свойства каждой композиции зависят от природы металла.

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