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ВЗАИМОДЕЙСТВИЕ КРЕМНИЯ С ГРАФИТОВЫМ КВАЗИМОНОКРИСТАЛЛОМ

М.С. Овчинникова, Н.И. Полушин, И.А. Бубненков, К.У. Кадыров

Мария Станиславовна Овчинникова*, Николай Иванович Полушин, Кадыр Узеирович Кадыров

Национальный исследовательский тухнологический университет «МИСиС», Ленинский пр., 4, Москва, Российская Федерация, 119991
E-mail: ovchmaria@mail.ru*, polushin@misis.ru, kadyrovku@mail.ru

Игорь Анатольевич Бубненков

Научно-исследовательский институт конструкционных материалов на основе графита «НИИграфит», ул. Электродная, 2, Москва, Российская Федерация, 111524
E-mail: iabubnenkov@mail.ru

Целью исследования, представленного в данной статье, является изучение кинетики пропитки графитового квазимонокристалла кремнием и изучение образования карбида кремния в результате жидкофазного взаимодействия кремния с квазимонокристаллическим графитом. Эксперименты проводились в реакционной камере установки высоковакуумной печи под давлением и нагреванием до температуры плавления кремния. Момент образовавшейся капли кремния на графитовой подложке фиксировался в зависимости от времени выдержки, затем проводился расчет радиуса капли с целью определения характера процесса смачивания. В соответствии с полученными данными измерялись геометрические параметры капли (высота, длина и угол смачивания). Показано, что увеличение времени взаимодействия квазимонокристалла с кремнием увеличивает межплоскостное расстояние между атомами углерода, что, по мнению авторов, связано с проникновением атомов кремния между плоскостями графита. Было также отмечено, что во время взаимодействия квазимонокристалла из графита с кремнием наблюдается интенсивный процесс снижения адсорбции (эффект Ребиндера), в результате чего кремний проникает в структуру углерода в направлении графитовых слоев. При взаимодействии кремния с квазимонокристаллом микрогруппировки исходного углерода мигрируют в расплав в результате процесса диспергирования. В статье представлены данные об экспериментах по смачиванию подложек квазимонокристалла кремнием с различными временами выдержки, изучены процессы на границе раздела полученных образцов. Из анализа экспериментальных данных о смачивании квазимонокристаллических подложек кремнием при разных временах выдержки была показана графическая зависимость радиуса и угла растекания образовавшейся капли металла во времени.

Ключевые слова: графит, квазимонокристалл, смачивание, эффект Ребиндера

INTERACTION OF SILICON WITH GRAPHITE QUASI-SINGLE CRYSTAL

M.S. Ovchinnikova, N.I. Polushin, I.A. Bubnenkov, K.U. Kadyrov

Maria S. Ovchinnikova*, Nikolay I. Polushin, Kadyr U. Kadyrov

National University of Science and Technology «MISiS», Leninsky pr., 4, Moscow, 119991, Russia
E-mail: ovchmaria@mail.ru*, polushin@misis.ru, kadyrovku@mail.ru

Igor A. Bubnenkov

State Research Institute of Structural Materials Based on Graphite, Electrodnaya st., 2, Moscow, 111524, Russia
E-mail: iabubnenkov@mail.ru

The aim of the study presented in this paper is to study the kinetics of the impregnation of a graphite quasi-single crystal by silicon, the study of the formation of silicon carbide in the liquid-phase interaction of silicon with a graphite quasi-single crystal. The experiments were carried out in the reaction chamber of the installation of a high-vacuum furnace under pressure and heating to the melting point of silicon. The moment of the formed drop of metal on the substrate was fixed, depending on the holding time with calculation of the droplet radius. According to the received data, the geometrical parameters of the droplet (height, length and contact angle) were measured. It is shown that an increase in the interaction time of a quasi-single crystal with silicon increases the interplanar spacing of carbon, which, in the opinion of the authors, is due to the penetration of silicon atoms between the graphite planes. It was also noted that during the interaction of a quasi-single crystal of graphite with silicon, an intensive process of adsorption decrease in strength (the Rehbinder effect) is observed, as a result of which silicon penetrates the carbon structure in the direction of the graphite layers. In the interaction of silicon with a quasi-single crystal, the microgroups of the original carbon migrate into the melt as a result of the dispersing process. The paper presents data on the experiments of wetting substrates of a quasi-single crystal with silicon with different holding times; processes at the interface between the obtained samples were studied. From the analysis of the experimental data on the wetting of the quasi-single-crystal substrates by silicon at different holding times, the dependence of the radius and the spreading angle of the formed metal drop on time were plotted graphically, and X-ray phase analysis was performed.

Key words: graphite, quasi-single crystal, wetting, Rehbinder effect

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INTRODUCTION

The physicochemical processes that underlie the melting of metals on a graphite substrate are the processes of interaction of a liquid metal with a carbonaceous substance that involve atomic dissolution and dispersal of the carbon component. Directly the process of interaction of metal with a substrate is preceded by wetting, spreading, capillary impregnation [1]. In parallel, the process of recrystallization of the disordered component proceeds. The most important factor in the course of these processes is the nature of the carbonaceous substance, which includes the crystal structure, porosity, etc. After the flow of the wetting and spreading process, the melt penetrates into the graphite through the capillary system. Surrounded by interlayers of the metallic phase under the action of the Rebinder effect, the macroparticles of the graphite phase pass into the melt. The most effective penetration of the metal melt into the porous system will occur with a decrease in the contact angle of wetting, and an increase in the effective radius of the capillary while maintaining the fluid properties unchanged.

The purpose of this study is to study the kinetics of impregnation of a graphite quasi-single crystal by silicon. Investigation of the formation of silicon carbide during the liquid-phase interaction of silicon with a graphite quasi-single crystal. A quasi-single crystal is a pyrographite heat-treated at high temperatures and pressures.

Thermomechanical treatment of pyrographite in the temperature range 2700-3000 °C with application of pressure 10-30 MPa leads to its recrystallization [2]. As a rule, thermomechanical treatment is carried out under the pressure of an inert gas, partially reducing sublimation of carbon at high temperatures. A quasi-single crystal consists of anisotropic mosaic blocks textured in a direction parallel to the basal planes. The structure of the initial pyrographite [3] affects the properties of the material after thermomechanical treatment. In all cases, this leads to a decrease in the size and partial disappearance of the growth cones in pyrographite. Investigations of the structural parameters and physical properties of a quasi-single crystal showed its high anisotropy, close to ideal graphite. Despite the approximation of a quasi-single crystal with respect to electrical resistance and thermal conductivity to an ideal graphite crystal, the presence of a mosaic block structure will facilitate the efficient dispersion of large structural fragments in the melt. The material is used in brake pads, missile nozzles, coating of combustion chambers of rocket engines, as a seal of graphite crucibles for melting high-temperature metals and refractories, etc.

The field of application of carbon materials is their use in the creation of composite materials with unique properties. Metals and alloys are used as a binder for such composites. Important tasks are to ensure excellent wettability of the carbonaceous material particles with a bond and strong adhesion of the cured metal to their surface. In addition, the technology for obtaining special grades of graphite, graphite for the synthesis of diamonds and continuous casting crystallizers is based on general scientific ideas about the interaction of these graphites with liquid metals.

EXPERIMENTAL TECHNIQUE

The experiments were carried out in the reaction chamber of a high vacuum furnace under pressure and heating to the melting point of silicon. The formed drop of metal on the substrate was fixed, depending on the holding time and calculation of the droplet radius. According to the received data, the geometrical parameters of the droplet (height, length and wetting contact angle) were measured. Then, the samples were analyzed on the Axio Observer A1m research inverted microscope from «Zeiss», specific surface energy was calculated and X-ray phase analysis was performed.

RESULTS AND ITS DISCUSSION

From the analysis of the experimental data on the wetting of the quasi-single crystal substrates by silicon at different holding times, the dependences presented below are constructed.

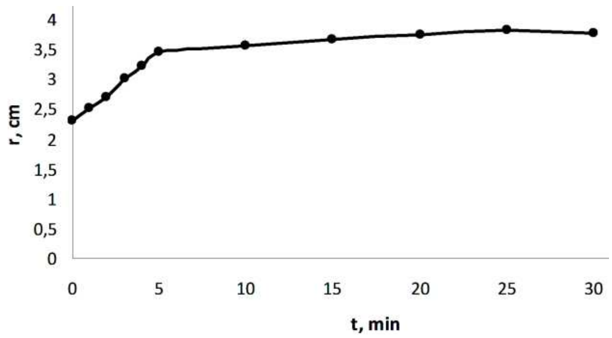

Fig. 1. The dependence of the radius of the silicon drop on the holding time for a quasi-single crystal

Рис. 1. Зависимость радиуса капли кремния от времени выдержки для квазимонокристалла

With an increase in the spreading area, which depends on the radius, the specific spreading surface will increase. From the dependence obtained, it can be concluded that the specific surface area of the spreading is increasing, which is one of the parameters, the quantitative evaluation of the spreading.

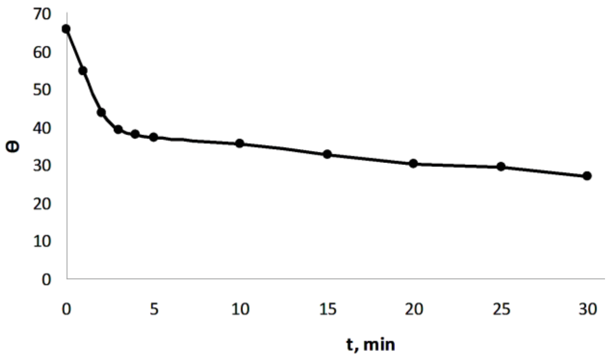

Fig. 2. The dependence of the contact angle of a silicon droplet on the holding time for a quasi-single crystal

Рис. 2. Зависимость угла смачивания капли кремния от времени выдержки для квазимонокристалла

The work of adhesion will increase with a decrease in the wetting angle, which indicates the intensive interaction of the contacting phases.

Fig. 3. Silicon droplet on a quasi-single crystal substrate (at a holding time of 1 min)

Рис. 3. Капля кремния на квазимонокристаллической подложке (время выдержки 1 мин)

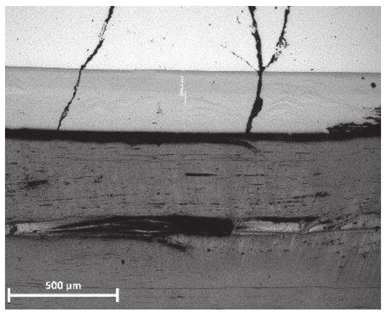

Fig. 4. Photographs of samples after experiments on wetting a quasi-single crystal with silicon (layer thickness is 264.3 µm, holding time is 1 min)

Рис. 4. Фотография образца после эксперимента по смачиванию квазимонокристалла кремнием (толщина слоя 264,3 мкм, время выдержки 1 мин)

Images of the contact site of the interphase interaction were obtained on the research inverted microscope. Microscopic images of the obtained sample after experiments on wetting of graphite substrates of a quasi-single crystal by silicon are shown in Fig. 4.

According to the photographs, the increase in the carbide layer of silicon and the ordered crystals with increasing time is clearly visible. This characterizes the increase in the interplanar distance of carbon, which is probably due to the penetration of silicon atoms between the graphite planes. According to the photographs, microgroups of the original carbon are transferred to the silicon melt as a result of the dispersion process.

CONCLUSIONS

It is shown that with an increase in the interaction time of a quasi-single crystal with silicon, the interplanar distance of carbon increases, which is probably due to the penetration of silicon atoms between the graphite planes. In the interaction of a quasi-single crystal of graphite with silicon, an intensive process of adsorption decrease in strength (the Rehbinder effect) is observed, as a result of which silicon penetrates the carbon structure in the direction of the graphite layers.

It is established that the interaction of silicon with a quasi-single crystal in the melt as a result of the dispersing process transforms the microgroups of the original carbon.

ЛИТЕРАТУРА

1. **Бубненков И.А., Кошелев Ю.И., Сорокин О.Ю., Чеблакова Е.Г., Полушин Н.И., Степарева Н.Н.** Оценка возможности применения углеродного наполнителя для изготовления силицированных изделий. *Новые огнеупоры*. 2011. № 12. С. 32-37.
2. **Фиалков А.С.** Углерод, межслоевые соединения и композиты на его основе. М.: Аспект Пресс. 1997. 718 с.
3. **McKinley I.M., Smith C.H., Ramsey P.G., Rodriguez J.I.** Pyrolytic graphite film thermal straps: Characterization testing. *J. Cryogenics*. 2016. V. 80. N 1. P. 174–180. DOI: 10.1016/ 2016.10.002

REFERENCES

1. **Bubnenkov I.A., Koshelev Yu.I., Sorokin O.Yu., Cheblakova E.G., Polushin N.I., Stepareva N.N.** Evaluation of the possibility of using carbon filler for the manufacture of silicized products. *Novye Ogneupory*. 2011. N 12. P. 32-37 (in Russian).
2. **Fialkov A.S.** Carbon, interlayer compounds and composites based on it. M.: Aspect Press. 1997. 718 p. (in Russian).
3. **McKinley I.M., Smith C.H., Ramsey P.G., Rodriguez J.I.** Pyrolytic graphite film thermal straps: Characterization testing. *J. Cryogenics*. 2016. V. 80. N 1. P. 174–180. DOI: 10.1016/ 2016.10.002

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