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RESEARCH OF METHODS OF OBTAINING CONTINUOUS FIBRES OF WHISKERS

Об'єктом цього дослідження були деякі методи отримання безперервних волокон ниткоподібних кристалів. Досліджено метод отримання таких волокон екструзією металоорганічного колоїдного розчину на прикладі принципової схеми проведення такого процесу. На прикладі іншої принципової схеми досліджено процес отримання волокон методом піролізу полімерних волокон. До проблемних сторін проведення обох процесів і використання таких методів слід віднести складність отримання потрібної конфігурації і орієнтації волокон ниткоподібних кристалів, а також недостатню досконалість апаратури і установок отримання таких волокон. При написанні роботи використовувалися різні методи наукових досліджень, такі як метод статистичного аналізу, метод аналізу результатів досліджень, гіпотетико-дедуктивний метод і метод узагальнення результатів. Проведені дослідження показали, що умови проведення обох процесів повинні строго контролюватися, особливо підйом температури. Показано, що застосовувані методи мають підвищену ступінь небезпеки. Обґрунтовано, що, незважаючи на дотримання усіх параметрів процесів, що проводяться, може бути не виключений той факт, що підсумкові волокна кристалів не матимуть потрібної орієнтації і конфігурації. В результаті проведених досліджень показано, що отримання безперервних волокон ниткоподібних кристалів методом екструзії металоорганічного колоїдного розчину досить ефективно з точки зору отримання підсумкового продукту потрібної орієнтації і необхідних параметрів. Ефективність цього методу може бути підвищена, якщо успішно використовувати процеси легування волокон з газової фази. Показано, що при проведенні досліджень отримання аналогічних волокон методом піролізу можна мати високу ефективність завершеного процесу при повному дотриманні параметрів його проведення і достатній досконалості устаткування. Досить істотним питанням під час використання саме цього методу є максимально ефективне проведення процесу натяжіння безперервних волокон ниткоподібних кристалів в процесі термообробки.

Ключові слова: *методи отримання ниткоподібних кристалів, безперервні волокна, металоорганічний розчин.*

1. Introduction

The first monograph, which was devoted specifically to the methods and techniques for obtaining whiskers, was the work [1]. Today it is possible to say with certainty that this monograph was the first monograph in the Russian literature and one of the first in the world, which would generalize the questions of the results of experiments, the methods used and the methods of research of whiskers. This was relatively recent and it should be noted that now the problem of studying the methods of obtaining whiskers continues to be quite relevant.

On this issue, most of the material still continues to be published in journals, conference proceedings and symposiums. Therefore, it is rather difficult to single out the main generalizing aspects of the processes and results of the studies carried out on the basis of a large number of sources of information.

The versatility and significant expansion of the range of use of various nanostructures is no longer in doubt. Sales volumes of nanotechnology products, where whiskers are widely used, already make billions of dollars (electronic devices, transistors, processors, ultraviolet and X-ray optics, etc.).

Therefore, the tendencies of a detailed study of the methods and methods for obtaining various groups of whiskers and their fibers become more and more widespread.

2. The object of research and its technological audit

The object of this research is some methods of obtaining continuous fibers of whiskers.

The method of extrusion of an organometallic colloidal solution, which under modern conditions is widely used to produce ceramic fibers that have a polycrystalline structure, has been considered. This method is analyzed on the example of a typical technological process, which is carried out according to the principle scheme for obtaining ceramic fiber of aluminum oxide.

Regarding the other scheme, the method of pyrolysis of a polymer fiber, which serves mainly for the production of carbon fibers, is considered. Considering the technological process of obtaining this type of fiber, it is stated that it is based on the thermal decomposition of such fibers with complete control of the process. The structure of the hydrocarbon fiber of the crystal is analyzed and its structure is analyzed.

The weak (problematic) sides of both ongoing processes when using these methods is the issue of control over the implementation of regulations and rather high risks of operations performed with their sufficient simplicity. It should be noted that the structure of the crystals obtained by these methods completely depends on the parameters of the regulation, which leads to the complexity of obtaining crystals of a certain structure and orientation.

3. The aim and objectives of research

The aim of research is reviewing the existing ideas about some methods of obtaining continuous fibers of whiskers.

To achieve this aim, it is necessary to solve such problems:

1. To investigate the method of extrusion of an organometallic colloidal solution, as well as the technology of carrying out this process and to determine the weak and strong sides of its conduct.

2. To investigate the method of pyrolysis of polymer fiber, to study the structure of carbon fiber, positive and problematic aspects of the implementation of the process regulation.

4. Research of existing solutions of the problem

As indicated earlier, at the end of the 20th century there were an extremely small number of generalized sources of information (monographs) on the research problem. The methods and techniques for obtaining various groups of whiskers were described and published rather disconnected. However, to say that there were no materials, of course, it is impossible. The problem was described, negotiated, examined and the ways of its solution were found.

As for whiskers, the complexity is not only their production and growing, but, most importantly, the obtaining of the desired result and the desired crystal structure, its necessary orientation. To obtain a crystal of the desired structure and the necessary orientation is almost 80% of the scientist's expenses, efforts and ingenuity [2].

Controlling the conduct of processes, the strict observance of all operations with any method of obtaining whiskers is the most important advantage of any method and technique used [3, 4].

In the first half of the 20th century, container methods of growing various groups of whiskers began to be applied, and magnetic fields were used to increase crystal growth, as is pointed out in [5–8].

Questions of «extension» or «stretching» of crystals, which were used in many methods (in particular, the Czochralski method) are described in [9–12].

The use of an electric field in the growth of whiskers and the processes of obtaining crystals from various groups of molten metals are described in detail in [13–15], which is generalized in [16, 17]. Although it should be noted that there were opponents of the application of methods of electrodeposition during the growth of whiskers, which, in particular, is indicated in [18].

The purity of the crystal itself has a great influence on crystal growth. When using, for example, the method of electron beam heating, this factor has a decisive influence [19].

Other methods of obtaining whiskers are also shown in the literature, for example, using arc furnaces [20, 21], using lasers [22] and induction heating [23, 24].

In [25–30], an analysis is made of methods for obtaining whiskers using chemical reactions for the reduction of halide metal salts.

In [31–34] summarizing materials of conferences are presented, where questions of the fields of application of whiskers are considered.

A certain generalized material on the analysis of the existing methods for the production of whiskers takes place in [35, 36], but with respect to the investigation of the various properties of whiskers, in particular:

- [37, 38] – on the investigation of the mechanical properties of whiskers;
- [39] – on non-traditional methods of growing whiskers;
- [40] – on the traditional methods of growing whiskers;
- [41] – on the methods of studying whiskers in a composite material;
- [42, 43] – on the properties and methods of obtaining whiskers of various groups (generalized literature review).

Thus, the results of the analysis lead to the conclusion that research on the existing methods for the production of whiskers and their fibers has received considerable attention. However, even today there are a fairly small number of systematized sources for this problem.

5. Methods of research

The following methods are used during the research:

- statistical analysis (to determine the dynamics of changes in approaches to the improvement of methods for obtaining continuous fibers of whiskers);
- analysis of research results (for determining positive changes and problematic issues of application of a method of obtaining continuous fibers of whiskers);
- hypothetical-deductive method (when acquainting the actual research material in the field of obtaining continuous fibers of whiskers, which additionally require in-depth analysis of additional sources of information);
- method of generalizing the results (to establish the general properties and trends characteristic of the methods of obtaining continuous fibers of whiskers).

6. Research results

6.1. Method of extrusion of an organometallic colloidal solution (suspension). It should be noted that this method is used to produce ceramic fibers that have a polycrystalline structure.

In general, the technological process for obtaining this group of fibers consists of the following operations:

1. Preparation of a colloidal solution of the corresponding compound.

2. Extrusion of the solution to form the desired continuous fibers of whiskers.

3. Remove of uncharacteristic compounds from the resulting fiber by firing. This operation as a whole contributes to the stabilization of the composition and structure of the obtained fiber of a whisker and creates the necessary degree of its compaction.

In the work on the example of the schematic diagram, the process of obtaining a ceramic fiber of an alumina (Fig. 1) is considered. For the production of this continuous fiber, a colloidal solution is initially prepared, as indicated in the first step, in this case a 50 % aqueous solution of aluminum formoacetate – $Al(OH)(CHO_2)(C_2H_3O_2)$.

After the preparation of such solution, it is evacuated, poured into a container (2) and at a pressure of 1–1.2 atm. nitrogen is supplied. Nitrogen is supplied from a cylinder (1). Further, the colloidal solution enters the pump (3), which serves to maintain the desired pressure throughout the pipeline. The pressure is fixed by the manometer (4). Purification from solid particles is carried out through the

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filter (5). Further, the purified colloidal solution enters the dies (7) through the pipeline.

To maintain the required viscosity of the solution, an isothermal bath (6) is created in the pipeline. The dies are 12 mm in diameter with a bottom thickness of up to 4–5 mm, where there are 12–15 holes with a diameter of 127 μm to form fibers. Fibers, passing through these dies, get into the air heated from the air heater (8), resulting in the evaporation of water and the formation of a sufficiently solid continuous fiber.

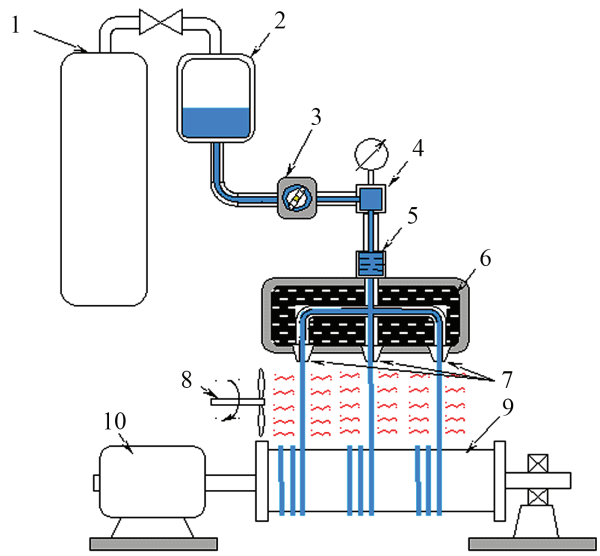

Fig. 1. Scheme of unit for the production of fibers of a polycrystalline structure: 1 – cylinder (with nitrogen); 2 – reservoir; 3 – pump; 4 – manometer; 5 – filter; 6 – isothermal bath; 7 – die; 8 – heater; 9 – drum; 10 – engine

The fiber then passes through the drum (9), winds on it, stretches, resulting in a change in the size of its diameter to 10–20 microns. The fiber is subsequently fired at temperatures up to 1500 °C. It should be noted that the process of heating the fiber is gradual and slow enough, so that the fiber effectively loses its organic component.

It should also pay attention to the fact that for this process, temperatures of up to 600–700 °C are sufficient. In the fiber structure, amorphous alumina remains.

Later, the fiber slowly heats up to 950 °C, which leads to the formation of the Al_2O_3 γ-modification. This modification later on, when heated to 1100–1200 °C and higher, turns into α-Al_2O_3 [44].

As can be seen from the description of the operation process of the above scheme, the main factor is the factor of a gradual increase in temperature and the tracking of the parameters for changing the orientation of the fibers.

6.2. Method of polymer fiber pyrolysis. This method also has certain directivity and serves mainly to produce carbon fibers. The material used in this method as a raw material is mainly synthetic or cellulose fibers. This type of fiber is used in sufficient quantities at textile industry enterprises. Less often, but still, how raw materials can be used and resins.

As in the first case, the fibers can also be obtained by passing through the spinnerets (pushing through them). However, the main condition is that the polymer must be in a permanently viscous state.

Considering the technological process of obtaining this type of fiber (carbon), it should be noted that it is based on their thermal decomposition with complete control of the process. This, first of all, is determined by the danger of the process. Heating temperature averages up to 3000 °C. The process medium is inert, resulting in the pyrolysis, decomposition and crystallization of carbon with the formation of C=C bonds.

Fig. 2 shows the carbon fiber, its structure. In general, it represents several hundred (sometimes thousands) fibrils that are aligned along the fiber axis [44].

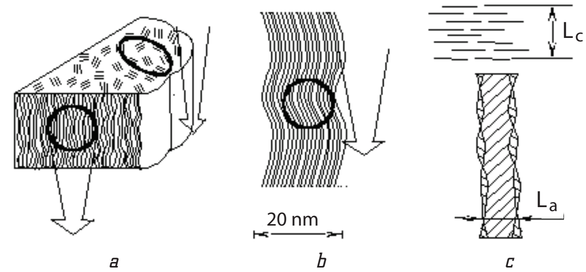

Fig. 2. Scheme of the structure of carbon fibers:
a – the general form of the fiber; *b* – longitudinal section of the fibril;
c – microfibrils (L_a and L_c – linear dimensions of microfibrils)

The structure of the carbon fiber itself is quite similar to the structure of the initial polymer. The fibril consists of ribbon-like layers of condensed carbon (Fig. 2, *b*). In Fig. 2, *c* shows that the microfibrils are separated by pores. At the same time, the orientation, both fibrils and microfibrils, is almost the same.

The process of formation of carbon fibers includes the following operations:

1. Heating to a temperature of 120–150 °C in order to remove water.
2. Subsequent slow heating to a temperature of 180–240 °C to remove the –OH groups and form C=C bonds.
3. Continuation of heating up to a temperature of 380 °C for the purpose of cleavage of cellulose rings
4. Further heating to a temperature of 650–700 °C to form graphite-like rings. At this stage, the threads are stretched to obtain the desired orientation of the crystals.
5. Graphitization process at temperatures up to 2000 °C.

However, it should be noted that not all processes of this method are of the same type and not all types of fibers are obtained in a similar way. Many of them must be subjected to endurance.

So, for example, during the heat treatment of polyacrynitrile fibers, the process of obtaining them looks like this:

1. Heating to 220 °C and holding for up to 20 hours.
2. Enough sharp increase in temperature to 950–1000 °C in an atmosphere of hydrogen and aging, as a rule, during the day.
3. Increase in temperature to 2000–2400 °C for 1.5–2 hours and the process of tension for 15–20 minutes with a pressure of 3–4 MPa.
4. Graphitization process for up to 20 minutes at a temperature of 2700–2900 °C.

Conclusions of a rather large number of experimental studies have shown that the issue of increasing the strength of carbon fibers during such process is quite promising.

7. SWOT analysis of research results

Strengths. As a result of using the method of extrusion of an organometallic suspension (colloidal solution), the strong points are certainly:

1) efficiency and ease of installation;

2) ease of carrying out the process of preparing a suspension from an organometallic compound;

3) invariance of the viscosity parameters during the process;

4) additional tension of the fibers leads to a decrease in the diameter of the holes in the die, which, in combination with the increase in temperature, facilitates the creation of various modifications of the fibers of the whiskers;

5) tensile strength parameters obtained with this method of obtaining fibers are sufficiently significant.

As a result of applying the method of pyrolysis of polymer fibers, the strengths are:

1) raw materials are cheap enough and at the same time efficient in use;

2) starting organic fibers are obtained by extruding through the dies of the corresponding polymer in the viscous flow state (not a complicated process);

3) structure of the crystal fibers varies due to a constant increase in temperature (uniformity of the process);

4) obtained carbon fibers have high mechanical properties at low density.

Weaknesses. The weaknesses of the research process include the following:

1) conditions for carrying out the processes must be strictly controlled, especially the time exponents of the temperature rise;

2) risks of the processes are significant;

3) structure of the obtained crystals is completely dependent on the parameters of the regulations, which leads to the complexity of obtaining crystals of a certain structure and orientation.

Opportunities. The studies of obtaining continuous fibers of whiskers by extrusion show that this method is quite effective in terms of obtaining the necessary configuration and modification of the fibers of whiskers. With a sufficiently controlled and precise observance of the parameters of the conducted processes, in most cases it is possible to preserve the desired properties and indices of the final product of a fiber of the whisker.

Threats. The threats are the complexity of obtaining the desired configuration and orientation of the fibers of threadlike crystals and the insufficient perfection of the apparatus and installations for obtaining such fibers.

8. Conclusions

1. A study of obtaining continuous fibers of whiskers by extrusion of an organometallic colloidal solution is made. It is shown that the use of this method is quite effective from the point of view of obtaining the final product of the desired orientation and the necessary parameters. The effectiveness of this method can be improved if the processes of doping the fibers from the gas phase are successfully used.

2. When carrying out the pyrolysis method for obtaining continuous fibers, it is shown that the application of this method can have a high efficiency of the completed process, with full observance of the process parameters, sufficient perfection of the equipment with sufficient simplicity and simplicity of the process. A fairly significant issue in carrying out this particular method is the maximum effective use of the tension process for continuous fibers of whiskers during heat treatment.

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