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ANALYSIS OF THE CURRENT STATUS OF THERMOELECTRIC MATERIALS AND TECHNOLOGY FOR OBTAINING AND MANUFACTURING HALF-ELEMENTS

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Abstract:

Objective. This article discusses in detail the materials that are of interest in the thermoelectric ratio. The most common members of the $A_2^V B_3^{VI}$ group in the periodic table of Mendeleev. The analysis of the current state of triple compound thermoelectric materials, which are the best materials for thermoelectric generators, as well as the methods and technology of production, the requirements for the technology of obtaining these materials, is carried out.

Methods. Experimental studies were carried out to determine the optimal concentration of current carriers of the required sign, by the method of formation of a non-stoichiometric chemical composition, or by the method of introducing additional dopants.

Results. To obtain a thermoelectric generator with high values of efficiency, as our studies show, the method of fusion under pressure of an inert gas [6-10] is the most effective, which differs in that melting is carried out in a crucible with a shutter. At the same time, the issue of container reuse is being addressed. For the manufacture of half-elements, cutting of solids plays an important role, which affects the energy parameters and efficiency.

Conclusion. The best and most effective way to obtain substances for the legs of a thermoelement is the method of fusion of the charge under the pressure of an inert gas. Thermoelectric semiconductor compounds obtained by this method have high values of thermoelectric figure of merit Z, reaching up to $3.0 \cdot 10^{-3}$ deg^{-1}, which makes it possible to create thermopiles with the best efficiency. The use of an electric spark cutting machine makes it possible to obtain thermopiles with minimal defects and the best quality.

Keywords: thermoelectric material, synthesis, doping, inert gas pressurization technology, half-elements, oxygen cutting, etching

Introduction. The most widely used as low-temperature thermoelectric materials are solid solutions of chemical compounds of the $A_2^V B_3^{VI}$ type from the elements of the fifth and sixth groups of the periodic system of D.I.Mendeleev. Let us agree to call the temperature interval 100-600 K. The natural boundaries of this interval are determined by the possibilities of using a class of substances whose initial components are bismuth, antimony, selenium and tellurium.

The operating conditions of thermoelectric materials in this temperature range are generally favorable. The absolute temperatures are relatively low, which significantly reduces the negative impact of such processes as oxidation, diffusion in the near-contact layers, volatilization of impurities and the main substance, etc.

Bismuth tellurium (Bi_2Te_3), selenium bismuth (Bi_2Se_3) and antimony tellurium (Sb_2Te_3) are of particular interest in thermoelectricity, since they are all used as initial components for creating efficient thermoelectric materials [1-4]. They are not used directly in their pure form, although they have rather high thermoelectric properties, especially when alloyed [4].

An analysis of the current state of thermoelectric materials shows that the best materials for thermoelectric generators are compounds obtained from semiconductor substances, which contain two or three components. Such semiconductors, especially the ternary compound, have a high thermoelectric figure of merit [4;5]. Obtaining chemical compounds and substances of a certain composition is the main task of synthesis. This is done using a variety of semiconductor technology techniques.

Synthesis is, as you know, obtaining complex structures and compounds from elementary and simple chemical compounds.

Methods. Various impurities have a very great influence on the thermoelectric properties of synthesized materials. Depending on the amount and type of impurities, the formation of various parasitic donor and acceptor levels and a non-optimal concentration of current carriers is possible. Therefore, to obtain a material with the same sign and the required concentration of current carriers, the components of the material should be cleaned before and after synthesis. A positive result of cleaning is a decrease in the degree of concentration of foreign impurities by one order of magnitude compared with the concentration of the main current carriers.

The greatest requirements are imposed on low-alloyed thermoelectric materials with a current carrier concentration of the order of ~10^{18} cm^{-3}.

The optimal concentration of current carriers of the required sign can be obtained by the formation of a non-stoichiometric chemical composition, or by the introduction of additional dopants.

In [6] a method is proposed for using nomograms to determine the optimal concentration of the dopant depending on the thermoelectric properties of the base. A number of batches of initial raw materials give bases, the thermoelectric properties of which do not fit within the limits of the nomogram [6]. In this case, it is not possible to obtain an alloyed material with the required properties only by changing the concentration of the introduced dopant.

It is known that some ratios of the feedstock give bases, the thermoelectric properties of which do not make it possible to obtain an alloyed material with optimal properties. Therefore, in order to correct the properties of the base Bi_2Te_3 and Bi_2Se_3, the parameters of materials obtained from different batches of raw materials were investigated. As a method for correcting the properties of the base, we used the introduction of superstoichiometric amounts of chalcogens into the charge. Tellurium and selenium were used as superstoichiometric chalcogens introduced into the charge.

According to the results of the experiments, for the n-type branches Bi_2Te_3-Bi_2Se_3, with the addition of 0.24 mol.% Te to the composition, an alloyed base was obtained with optimal values of its thermoelectric properties σ = 585 $Ohm^{-1}\cdot cm^{-1}$, α = 204 μV/deg., in figure 1. The optimal concentration of selenium added to the components was 0.12 mol.%, their thermoelectric properties σ= 580÷600 $Ohm^{-1}\cdot cm^{-1}$, α= 204÷194 μV/deg. (Fig. 2) [5;7;10].

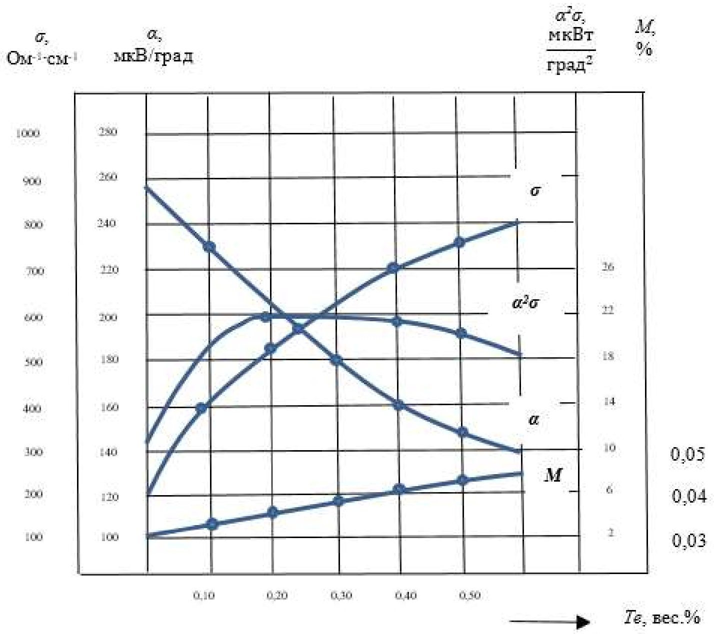

Fig. 1. Dependence of the indices σ, α, $\alpha^2\sigma$, and M on the Te concentration [5;7;10]

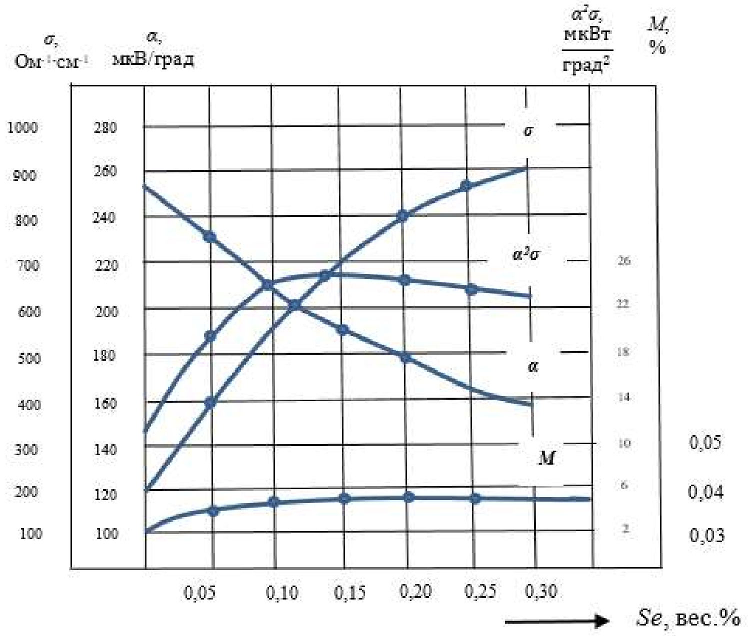

Fig.2. Dependence of the indices σ, α, $\alpha^2\sigma$ and M on the concentration Se [5;7;10]

The last stage is the manufacture of thermoelement legs. However, this stage sets the task of obtaining a thermoelectric material with strong uniform and thermoelectric properties. After that, the materials are subjected to machining and polishing.

Discussion. It is known that it is not always possible to directly use purified, synthesized and alloyed thermoelectric material for the manufacture of thermoelement legs. Formed thermal cracks, porosity and other defects do not allow obtaining thermoelement legs of the appropriate size by mechanical processing of the material. In such cases, the physical and mechanical characteristics do not meet the requirements. In this regard, there is a need to obtain monolithic and defect-free samples. They should be close in shape to the shapes of the thermoelement legs, the required design, and should be subject to minimal mechanical processing.

Such a technological process is carried out by the developed methods for the manufacture of FC branches:

- method of powder metallurgy;
- metallurgical methods;
- method for obtaining single-crystal samples.

The most common methods among powder metallurgy methods are the cold pressing method followed by heat treatment and the hot pressing method. Among the metal methods, the most common is the method of melting, subject to the conditions of directional crystallization. The third method, the method of obtaining single-crystal samples, is the most difficult. However, they are successfully used to obtain thermoelectric materials with the best thermoelectric properties compared to other existing ones. These methods include the Czochralski, Bridgman and various types of zone melting.

Results. To obtain a thermoelectric generator with high efficiency values, as our studies show, the method of fusion under pressure of an inert gas [8-12] is the most effective, which differs in that melting is carried out in a crucible with a shutter. At the same time, the issue of container reuse is being addressed. The fact is that after melting, the material, after light tapping, is easily removed from the overturned crucible without violating its integrity and the integrity of the "float". In this way, a large amount of expensive quartz can be saved.

Upon reaching the required temperature, an exposure is made during which the alloy is fused and homogenized. After holding the alloy at the specified temperature, the furnace is turned off, and the sleeve is cooled and the ingot is unloaded after the furnace has completely cooled to room temperature.

For the manufacture of half-elements, cutting of solids plays an important role, which affects the energy parameters and efficiency [12-20]. The existing various solid cutting methods such as oxy-fuel cutting, shielding gas cutting, precision cutting, micro-beam oxy-fuel cutting, flame gouging, iron powder feeding cutting, arc cutting, high-speed plasma cutting, and even laser cutting, for one reason or another, are not quite suitable for cutting semiconductor thermoelectric materials.

Even if some of the methods listed above are used, then one can see the roughness of the performed process, or, a change in the chemical composition of substances, the insufficient accuracy of the geometric dimensions of the half-elements. Our experience and analysis shows that the best option for using installations for cutting thermoelectric materials of a triple compound fused under inert gas pressure is the use of electric spark cutting installations.

At the same time, the substance from which the half-elements are cut is securely and firmly fixed to the moving parts of the installation. For this, special supports are used to fasten the material. Fastening is carried out by soldering. Soldering the substance on the stand can be done with bismuth tin solder (58 Bi, 42 Sn). This solder has a melting point of 136°C. The authors of [14] propose the use of BiSnSb solder with a melting temperature of 140°C. The soldering material we recommend is very suitable for heat sensitive parts in the semiconductor industry.

After cutting, the half-elements are etched. To ensure high-quality etching of semiconductor materials, their surface is thoroughly cleaned. This process is important because contaminated areas are not susceptible to etching. To prepare the surface for etching, you can use degreaser bottles, medium-hard industrial brushes (natural bristles), and spare degreaser bottles.

If the etching operation is carried out over a small number of half-elements of thermoelements (FCs), then it is sufficient to use a fluoroplastic cup with an etching mixture (of course, the mixture must be specially prepared!). In the case of serial production, pickling devices with and without heating should be used. Water is known to be used to terminate the etching reaction. Therefore, for this purpose, the use of washing machines with a manual or mechanical feed system is recommended. Etched and washed plates or crystals are laid out in a clean vessel.

Conclusion. The best and most effective way to obtain substances for the legs of a thermoelement is the method of fusion of the charge under the pressure of an inert gas. Thermoelectric semiconductor compounds obtained by this method have high values of thermoelectric figure of merit Z, reaching up to $3.0 \cdot 10^{-3}$ deg^{-1}, which makes it possible to create thermopiles with the best efficiency. The use of an electric spark cutting machine makes it possible to obtain thermopiles with minimal defects and the best quality.

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