Diffusion welding in gradient heatmetry

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Gradient heatmetry allows you to record, process and analyze pulsations of heat flux, which is of paramount importance in the research of gas flow around bodies, in the study of complex heat transfer, etc. Gradient heat flux sensors (GHFSs) made of materials with anisotropy of thermal and electrical conductivity and Seebeck coefficient have significant advantages. It is especially important that GHFSs have an abnormally high speed: their time constant has a level of \(10^{-8}\) ... \(10^{-9}\) s, which is 3 ... 5 orders of magnitude less than that of the best world analogues. The amount of natural anisotropic metals is small, and it is impossible to influence their properties; in addition, the limitations imposed by their thermal stability are fundamentally unavoidable. For example, GHFSs based on bismuth single crystals of 0.9999 purity have a high and temperature-independent volt-watt sensitivity; however, their field of application is limited by the melting point of bismuth (271°C). Therefore, there is obvious interest in artificial anisotropic materials.

To create an artificial anisotropic material, it is required to provide a set of properties that determine the functionality and reliability of the GHFS:

1. Anisotropy of thermal conductivity in two mutually perpendicular directions, attainable with significantly different thermal conductivity of the composite components.
2. Working temperature up to 1300 K and higher.
3. Chemical resistance in various environments.
4. Guaranteed and acceptably strong physical contact in the joint zone of the composite components.
5. Uniform planar and shallow in bedding from the interface front of volumetric interaction, providing a minimum change in the electrical properties of the components of the composite in the joint zone.

Such a composite – a laminated composite material (LCM) – can be obtained by diffusion welding. It allows varying technological parameters (pressure, time, temperature, degree of vacuum) within a wide range, and in the case of a developed and correctly activated surface of the materials to be welded, it guarantees the formation of a reliable connection while maintaining high physical properties of the components to be welded.
In the process of diffusion welding, a multilayer anisotropic bar was obtained. After cooling, it was cut into plates located at an angle of 20 ... 45° to the working planes, and then switching leads were attached.

The first composite materials for creating GDTP were LCM nickel + steel 12Cr18Ni9Ti, nickel + steel 65Cr13, chromel + alumel, iron + constantan, and copper + molybdenum. These GHFSs are efficient up to a temperature of 1300ºK. The volt-watt sensitivity of some GHFSs at a temperature of about 300 ºK is given in table 1.

### Table 1. Volt-watt sensitivity of GHFSs at a temperature of about 300 ºK.

| LCM                         | Sensitivity, mV/W |
|-----------------------------|-------------------|
| nickel + steel 12Cr18Ni9Ti  | 0,40              |
| chromel + alumel            | 0,35              |
| copper + molybdenum         | 0,02              |

For further research, we chose LCM nickel + steel 12Cr18Ni9Ti and chromel + alumel. The analysis of the distribution of elements in the joint zone was carried out on a Camebax-Microbeam microanalyzer manufactured by Cameca (France). Analysis accuracy is of 5%, spatial resolution: transverse (dia of the analyzed area) is of 1.5–2.5 µm; longitudinal (in depth) is of 0.7 µm.

The microstructure of the LCM nickel + steel 12Cr18Ni9Ti is shown in Figure 1. The results were obtained by electronically scanning the boundary perpendicular to it at a magnification of ×400 (a) and ×2000 (b). The width of the diffusion zone for the studied LCM is of about 1 ÷ 5 µm with a spread of 1 µm, which ensures the required strength of the joint and does not affect the initial materials by 90 ... 95% of their thickness.

Figure 2 shows the dependence of the volt-watt sensitivity of the described GHFs on temperature. Composition (a), in which the curves are monotonic, has an advantage.

The calculated volt-watt sensitivity of the GHFS depends on the accuracy with which the properties of the components are specified, but their spread in the literature often exceeds 10 ... 20%. The resulting significant difference between the calculated and experimental curves in figure 2, b indicates the priority of the data obtained during the calibration of the manufactured GHFSs.

Below the results of the calibration of GDTP made from the compositions nickel + steel 12Cr18Ni9Ti and chromel + alumel are shown (table 2).
Figure 2. Volt-watt sensitivity of GHFS: a – chromel + alumel; b – nickel + steel 12Cr18Ni9Ti. The numbers indicate: 1 – calculation results; 2 – experimental curves.

Table 2. The results of the GHFSs calibration.

| Reference temperature, °C | Heat flux per unit area, W / m² | Volt-watt sensitivity, mV / W, for compositions | Notes |
|---------------------------|---------------------------------|-----------------------------------------------|-------|
|                           |                                 | nickel + steel 12Cr18Ni9Ti | chromel + alumel | test time, s | with points processed |
| 104                       | 2600                            | 1,120                          | 0,599              | 3170         | 6340                  |
| 206                       | 5690                            | 1,620                          | 0,562              | 572          | 1140                  |
| 256                       | 9670                            | 1,400                          | 0,461              | 200          | 400                   |
| 336                       | 15200                           | 1,180                          | 0,443              | 297          | 594                   |
| 380                       | 22500                           | 0,844                          | 0,422              | 281          | 562                   |

Figure 3. Calibration curves for GHFSs: 1 – nickel + steel 12Cr18Ni9Ti; 2 – chromel + alumel.
From the graph (figure 3) it can be seen that in the temperature range of 250 ... 400 °C, which is of greatest interest for heat metering in aircraft engines and on the surface of streamlined airfoils, the characteristic of GHFS made of chromel-alumel composition is monotonic, but the sensitivity of sensors based on LCM nickel + steel 12Cr18Ni9Ti is higher almost an order of magnitude, which allows you to prefer this composition.

It was also possible to use permeable materials as layers: fibrous, mesh, with regular perforation, etc. Figure 4 shows the structure of LCM nickel + steel 12Cr18Ni9Ti (mesh).

**Figure 4.** a) – the sample (scale in mm), b) – microstructure of LCM nickel + 12Cr18Ni9Ti (mesh).

In diffusion welding, the intersections of the mesh threads are welded in the contact zones, and after cutting, the GHFS becomes permeable to liquid or gas flows, and the resistance does not exceed tens of Pa / mm (and can be regulated by the selection of the mesh, the angle of inclination when cutting the bar, and the GHFS thickness). The interpretation of the signal of such a GHFS is a separate problem, but its application in study of heat transfer in systems for blowing a cooled medium, gas curtains, etc. will give clear and unparalleled benefits.

Of particular interest is the use of semiconductor + metal and semiconductor + conductor compositions, since the values of the Seebeck coefficients in semiconductors are an order of magnitude or more higher than the level typical for metals.

Analysis of the literature has shown that sufficient attention has been paid to the preparation of a joint of a single silicon wafer with an aluminum foil; however, the preparation of LCM based on these materials has not been described. By the method of diffusion welding in air (at the temperature of 820 K and holding for 3600 s), it was possible to obtain a GHFS based on silicon + aluminum composition. The high solubility of aluminum in silicon, traditionally used in nonferrous metallurgy, played a positive role, providing a diffusion connection with a transition zone width of 5 ... 15 µm.

It was found that in order to increase the strength of the LCM, welding should be carried out immediately after the activation treatment. An increase in the roughness and (to a lesser extent) microhardness of the surface layer of a single crystal of silicon reduces the strength of the welded joint.

No less promising is a composition of alternating silicon layers with n- and p-conductivity. We used an aluminum foil of 0.05 mm thickness as interlayers between the silicon wafers and, with the described technology, we obtained a layered n- silicon + p-silicon composite. The sensitivity of such GHFSs is almost twice the level reached by GHFS silicon + aluminum, and their thermal stability is limited by the softening temperature of silicon (1100 K), however, poor reproducibility of the properties of LCM was revealed, which is largely associated, first, with the method of manufacturing a silicon single crystal (crucible-free zone melting or pulling the crystal out of the
melt by the Czochralski method), and, secondly, with the methods of abrasive processing of a single crystal. Both difficulties need to be addressed.

It is important that with the advent of high-temperature GHFSs, both the capabilities and the ideology of gradient heatmetry in aviation and other power plants change significantly.

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