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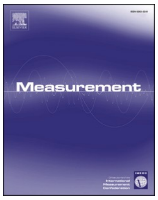

Method for evaluating the temperature of amorphous ferromagnetic microwires under Joule heating

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ABSTRACT

The results of comparative studies of the electrically conductive properties of $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ glass coated amorphous microwires obtained during their heat treatment in a conventional furnace and by the Joule heating method are presented. The fully crystallized microwire dramatically changes its electrically conductive properties. We found that the crystallized microwire has a temperature coefficient of resistance, $\alpha = 315*10^{-6}$ 1/ °C. The crystallized microwire was used as a reference resistance thermometer under Joule heating for determining the temperature of the microwire as a function of the applied thermal power *T(P)*. This dependence obtained was used to determine the temperature dependence of the resistance $R_M(T)$ of other microwire samples in an amorphous or partially crystallized state of the same series. The proposed method allows to select thermal modes during Joule annealing of microwires and to compare the resistive, magnetic and structural-phase properties of microwires after thermal effects.

1. Introduction

The glass coated amorphous ferromagnetic microwires produced by rapid melt-quenching using the Taylor-Ulitovsky method have unique combinations of magnetic and strength characteristics [1,2]. The Co-rich amorphous microwires are characterized by low values of coercive force and demonstrate the giant magnetoimpedance (GMI) effect with a quite large GMI-ratio [3,4]. The microwires can be used as sensitive elements in magnetoimpedance sensors [5,6] and as tips in scanning magnetometry devices [7,8].

One of the ways for improving the magnetic properties of amorphous ferromagnetic microwires after their manufacture is thermal treatment with current (Joule heating). With this treatment, a current of a given value is passed through the sample for a certain period of time. The influence of Joule heating on the magnetoimpedance effect and magnetostrictive properties is well known and studied in detail [9–11]. The behavior of amorphous microwires during heating and cooling can be studied, for example, by measuring their resistance [12,13]. In the process of heat treatment by the Joule heating method, an important task is to ensure the necessary temperature regimes for heating and the determination the microwire temperature. The solution to this problem is in finding the form of the dependence of the microwire temperature on the heating power [14]. In the work [15], a correlation between the average temperature of a microwire and the heating current density was obtained by means of comparing the temperature dependence of the saturation magnetization of two samples, one of which was subjected to Joule heating and another one to conventional annealing in a furnace. The theory of thermal effects of electric current in amorphous microwires [16,17] confirms the fact that thermal heating is practically uniform throughout the entire volume of a microwire under normal experimental conditions. The equilibrium temperature is reached as faster as higher is the current value, and the temperature distribution is approximately constant in the cross section of the microwire. The calculation formulas were experimentally verified by magnetic measurements of amorphous microwires with a known Curie temperature.

In real experiments, the existing models [17,18] allow one to establish the correspondence between the transmitted current and the heating temperature only approximately because of the wide scatter of the initial values and temperature coefficients of microwire resistances. Changes in the structure of the microwire that occur during heating are

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reflected in changes in resistance and depend on the magnitude and time of thermal exposure. The temperature dependence of the resistance during the first heating is nonlinear due to irreversible structural-phase transformations in the microwire that leads to changes in the initial values of the resistivity.

Direct contact measurements of the microwire temperature under Joule heating by using resistance thermometers or non-contact measurements by pyrometric methods have a large error because of small diameters of amorphous microwires. It was recently proposed [19] to use an additional microwire with a known temperature coefficient of resistance as a resistance thermometer for the experimental determination of the microwire temperature during Joule heating. The proposed method is based on the fact that if the length, cross-sectional area, specified power and heating conditions for two microwires of the same type are the same then the temperature of the microwire used as a resistance thermometer will be equal to the temperature of the microwire under study.

This paper presents the results of a comparative study of the properties of $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ glass coated amorphous microwires during heat treatment in a conventional furnace and by the Joule heating method. It was shown that the electrical resistance of the microwire can irreversibly change during annealing and that these changes can serve as an indicator of the microwire state. A fully crystallized microwire is characterized by a linear temperature dependence of resistance $R_M(T)$. We use this dependence to determine the temperature of microwires of the same series during Joule heating. The obtained temperature dependences of the resistance changes will make it possible to compare the magnetic and structural-phase properties of microwires after a certain heat treatment.

2. Experimental details

During investigations, we used pieces of glass-coated amorphous microwires of $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ composition [12,13,20,21] produced by Taylor-Ulitovsky method. The microwire samples had an outer diameter $D = \sim 28.5$ μm, a core diameter $d = \sim 13.5$ μm, and a length $l = 100$ mm. The ends of the samples were pre-stripped from the glass shell for providing electrical contact with a special holder. We have measured over 20 microwire samples. The values of their resistances at room temperature were in the range of 830 ± 30 Ohm. Samples with resistance in the range of 830 ± 10 Ohm were selected for further measurements.

The characteristic temperatures and the extent of crystallization were found using differential scanning calorimetry (DSC) and standard IT applications. The DSC measurements were performed with DSC 204 F1 Netzsch calorimeter in Ar atmosphere at the temperature range from 300 °C to 600 °C and with a heating rate of 10C/min. The sample masses were 3–10 mg.

A measuring system based on a Wheatstone bridge was used to heat the microwire samples and continuously measure their resistances during annealing. The general functional diagram of the installation is shown in Fig. 1. Sample of microwire with resistance R_M was fixed on a special holder and connected to one of the arms of the Wheatstone measuring bridge formed by precision resistors R_1 and R_2 and a resistor bank R_{RB}. The resistor R_1 had a high enough resistance ($R_1 \gg R_M$) so that the current I_1 flowing through it was small compared to the current I_M flowing through the microwire ($I_1 \ll I_M$). At the same time, the resistor R_2 had a low resistance ($R_2 \ll R_M$) so that the main thermal power was released on the resistance of the microwire R_M. The measuring system allows the microwire heating both in a standard furnace and in the Joule heating mode with direct current.

The bridge scheme was powered by a programmable DC voltage source in the voltage range U from 3 to 80 V that provided the current I_M with an amplitude up to 80 mA and thermal power P_M up to 5 W through the microwire. The direct current caused the heating of the microwire sample, which resulted in its resistance variation and an appearance of corresponding voltage of the bridge unbalance ΔU. We controlled a bridge supply voltage U, a voltage across the resistor R_2, and a voltage of the bridge unbalance ΔU on the diagonal of bridge during our measurements. Initially the minor voltage $U_0 = 3$ V was applied to the bridge and the bridge balance condition $\Delta U = 0$ was satisfied by using the selection of the resistor R_{RB} of resistor bank. The initial microwire resistance at room temperature R_{20} was found from the equation $R_{20} = R_2 \cdot R_1 / R_{RB}$. During heating, the thermal power $P_M = I_M^2 R_M$ released in the microwire led to heating and a corresponding change in its resistance R_M. The current I_M was determined by the voltage across the resistor R_2. The resistance R_M was calculated using the measured time series of U and ΔU [12]. To compare the resistance variations of different microwire samples, all results were presented in the normalized units with respect to initial resistance of the microwire sample at room

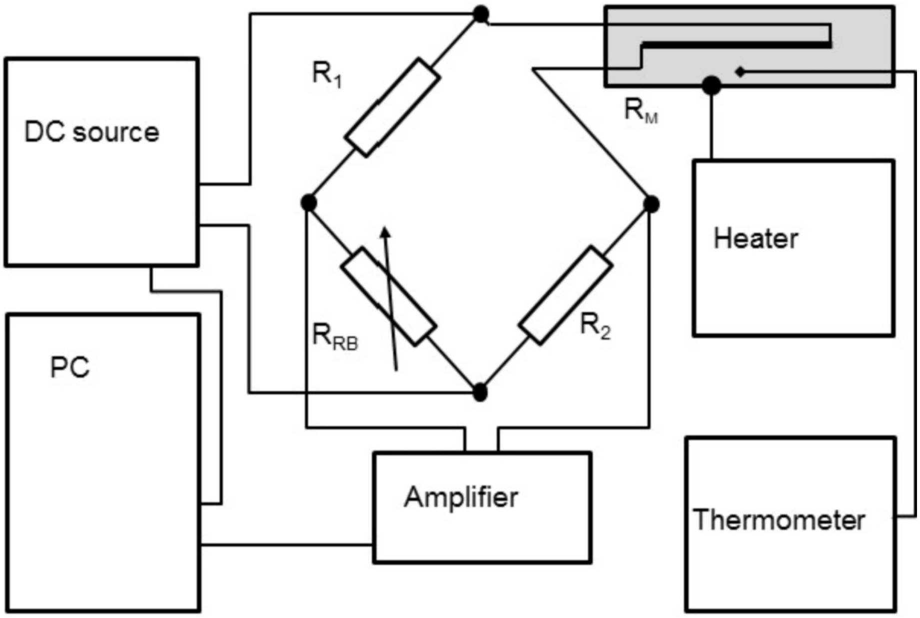

Fig. 1. Functional diagram of a measuring system based on a Wheatstone bridge for heating microwires and measuring their resistance during annealing.

temperature.

To determine the measurement errors of the measuring system, we carried out measurements with calibration resistors in the range from 780 Ohm to 880 Ohm and at a bridge supply voltage from 3 V to 10 V. For this, we used an additional precision resistor bank, with an accuracy of setting the resistance value up to 0.01 Ohm. It was found that the developed bridge circuit provides an accuracy of resistance measurements within ± 0.1 Ohm.

During the microwire annealing in a standard furnace, the temperature in the heating zone was increasing to 600 °C, after which the heater was turned off and cooling occurred due to the natural cooling of the furnace. Temperature control was carried out using a thermocouple. The temperature measurement error was within $\pm\ 0.0075 \times T$ of the measured temperature value. To register changes in the resistance of the microwire, a small dc voltage ($U = 3$ V) was applied to the bridge circuit, and the bridge unbalance voltage ΔU was continuously recorded. The characteristic time for a complete measurement cycle was 8 h.

During annealing by the Joule heating method, the furnace heater was turned off. A linearly increasing voltage U was applied to the bridge circuit, and the bridge unbalance voltage ΔU was continuously recorded. The characteristic time for a complete measuring cycle was 1 h. The process of annealing of microwire samples usually consisted of three stages: heating, holding, and cooling. The duration of each stage was usually 20 min. At the first stage, the heating power of the microwire increased linearly at a constant rate from zero to the selected maximum value. At the second stage, the microwire was kept at a constant maximum value of the specified power. At the third stage, the power also decreased linearly at a constant rate from the maximum value to zero, and the microwire was cooled. The main recorded parameters were the magnitude of the change in the resistance R_M of the microwire relative to the initial value of the resistance of the microwire R_{20} at room temperature and the power P_M released in the conductive core of the microwire during the current I_M flow.

3. Result and discussion

3.1. Furnace measurements

The DSC curve of the microwire under study is shown in Fig. 2. This DSC curve was used to determine the temperature of 514.7 ^{0}C for the onset of crystallization of the microwire, and to establish that the crystallization process at temperatures above 600 °C is completed. Two peaks corresponding to a two-stage crystallization process were observed at temperatures of 525 °C and 560 °C, respectively.

After DSC measurements, temperature measurements of the microwire resistance were carried out with direct heating in the furnace. The microwire was heated in the temperature range of 20–600 °C at the same rate of 10 °C/min as in the case of our DSC analysis. Using the measured time series of U and ΔU as the base, our computer program defined a temperature dependence of the microwire resistance R_M during direct heating in the furnace and calculated the regression line equation for a set of experimental points of the linear part of the data. The errors in the initial measurements of resistance were ± 1 Ohm. To compare the resistance variations of different microwire samples, results were presented in the normalized units with respect to initial resistance of the microwire sample at room temperature.

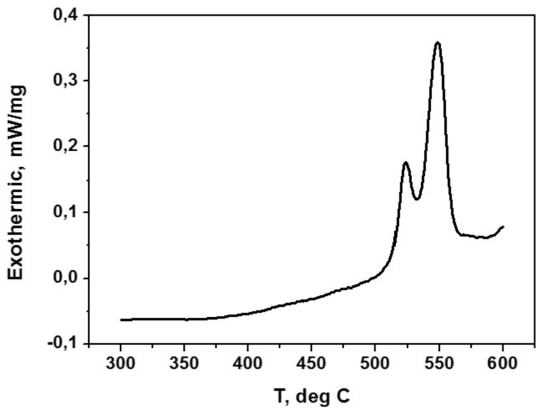

Fig. 2. DSC curve of a microwire with the composition $Co_{69}Fe_4Cr_4Si_{12}B_{11}$.

Fig. 3 shows the temperature dependence of changes in the normalized resistance of a microwire sample heated in a furnace. As can be seen, the normalized resistance values of the microwire vary within 1% of the initial value in the range from room temperature to ~ 500 °C. In the temperature range of the two-stage crystallization process, the normalized resistance undergoes abrupt changes. The rise in resistance, a sharp drop, and a new slow growth correlate well in temperature with the first and second crystallization peaks on the DSC curves. After reaching a temperature of 600 °C and the completion of the crystallization process, the temperature dependence of the normalized resistance changes significantly. The resistance decreases linearly during cooling, and at room temperature its value decreases by 13% in comparison with the initial value. Repeated temperature measurements of the same sample after cooling showed that the slope of the normalized resistance of the microwire coincides with the slope of the lower branch of the R_M/R_{20} dependence shown in Fig. 3. The slope of the temperature dependence of the resistance determines the value of the temperature coefficient of resistance (TCR) of the crystallized microwire. In our case, the TCR value was: $\alpha = 315{*}10^{-6}$ 1/°C.

3.2. Joule heating annealing.

During the Joule heating of the microwire sample, its temperature will be determined by the thermal power P_M released in the conductive core with an active resistance R_M at the current heating current I_M, $P_M = I^2_M{\cdot}R_M$. As shown in Section 3.1., the microwire heating can lead to irreversible changes in its resistance. Nevertheless, if the released thermal powers are equal, then the temperatures of microwire samples with different resistance values will also be the same. Therefore, when processing the results of Joule annealing of the microwire, the P_M power released in the microwire during the current flow was chosen as the main parameter.

By analogy with measurements in a furnace, we defined the microwire resistance R_M and the thermal power P_M, using the measured time series of U and ΔU. Fig. 4 shows the dependence of the normalized resistance of the microwire on the power released in it during the first complete annealing. The upper part of the curve in Fig. 4 reflects the

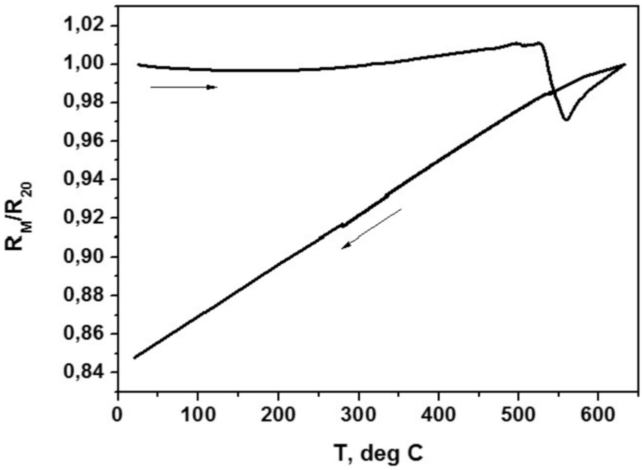

Fig. 3. Temperature dependence of the microwire normalized resistance during direct heating in the furnace. TCR value $\alpha = 315{*}10^{-6}$ 1/°C.

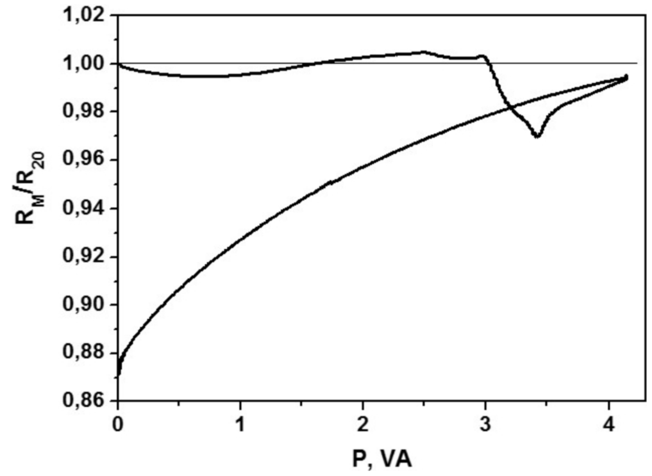

Fig. 4. Dependence of the microwire normalized resistance on the applied power during the first full Joule heating.

heating of the microwire with an increase in the applied power from zero to a maximum value of 4.2 W. At low power values of 0.0–0.8 W, a slight decrease in the normalized resistance is observed. With a further increase in power, it slowly increases. In the power range from 3.0 W to 3.4 W, a sharp drop in normalized resistance is observed, which is then replaced by it increase up to the maximum value of the applied power, 4.2 W. Holding the microwire for 20 min at a maximum heating power of ~ 4.2 W does not lead to additional changes in the value of its resistance.

The lower part of the curve in Fig. 4 corresponds to the cooling stage. With a decrease in the applied power, the resistance of the microwire decreases monotonically. The total value of the normalized resistance change after cooling to room temperature is ~ 13% with respect to the initial value. As expected, the changes in the microwire normalized resistance during annealing in a conventional furnace and during Joule heating correlate well with each other.

It is important to note that the temperature dependence of the microwire resistance that has undergone heat treatment at high (more than 4.0 W) heating powers stabilizes and does not change during subsequent heating and cooling cycles [12]. This allows crystallized microwires to be used as resistance thermometers after determining the microwire TCR.

3.3. Dependence of the amorphous ferromagnetic microwires temperature on the annealing power.

Using the TCR value obtained during annealing in the furnace, we converted the relative changes in the resistance of the crystallized microwire (the falling part of the curve in Fig. 4) from the applied power into the temperature T in accordance with the formula:

$$T = [R_M/R_{20} - 1]/\alpha + T_0 \tag{1}$$

where R_M is the resistance of the crystallized microwire during Joule heating, R_{20} is the resistance of the microwire at room temperature, $\alpha = 315{*}10^{-6}$ 1/°C is the microwire TCR obtained from the temperature dependence of annealing in the oven, T_0 is the room temperature (20 °C). The relationship between the power P_M released in the microwire from the heating current and its temperature T can be converted by means of a simple nonlinear regression into an analytical expression in the form of a polynomial of the n-th degree (calibration characteristic). The calibration characteristic of the temperature dependence on the heating power of the studied microwire $T = f(P_M)$ found in our case has the form:

$$T = 30.62 + 313.7{*}P_M - 73.71{*}P_M^2 + 7.793{*}P_M^3 \tag{2}$$

Graphs of the dependence of the microwire temperature on the heating power plotted with the use of Eq. (1) and based on the calibration characteristic (2) are shown in Fig. 5.

The calibration characteristic $T(P_M)$ shown in Fig. 5 with a solid line, is in good agreement with the experimental curve. However, the resulting errors in determining the temperature is ± 3 °C.

Eq. (2) actually reflects the heat balance equation and characterizes the redistribution of heat released from the heating power P_M into two parts: a) heat released in the microwire and b) heat dissipated as a result of heat exchange with the environment. As the temperature of the microwire increases, the amount of heat dissipated to the environment increases and the growth of the $T(P_M)$ curve slows down as the heating power increases.

Due to the relatively low thermal conductivity of air, the total temperature settling time is of the order of seconds. For a steady temperature, the amount of heat released in the microwire is equal to the amount of heat dissipated from its surface due to heat transfer. Therefore, the heating temperatures of two samples of microwires from the same series, with equal geometrical dimensions and heat transfer conditions, will be equal at equal heating powers. This allows a fully crystallized microwire to be used for obtaining a reference dependence of the microwire temperature on the heating power for other microwire samples of the same type and length. The resulting calibration curve of the dependence of the heating temperature on the heating power can be used to assess the temperature modes of heating and the associated effects of electrical and magnetic transformations in the microwire.

Thus, the use of crystallized microwire makes it possible to indirectly determine the temperature of microwire samples of the same type under Joule heating. For this purpose, a crystallized microwire with a known TCR must be placed under specified conditions during calibration tests and the $R_M(P_M)$ dependence to be measured during Joule heating. Using Eq. (1) and a polynomial approximation of type (2), one can establish the dependence $T(PM)$. This dependence is a calibration characteristic for determining the temperature of the same series of microwires under the same conditions during their Joule heating.

3.4. Temperature dependences of microwire resistance

Analytical dependence (2) was used in data processing after Joule heating to convert the thermal power applied to the microwire into the appropriate temperature. As an example, Fig. 6 shows the temperature dependences of the resistance of the same microwire, obtained during several successive Joule heating cycles. Each measurement cycle of 60

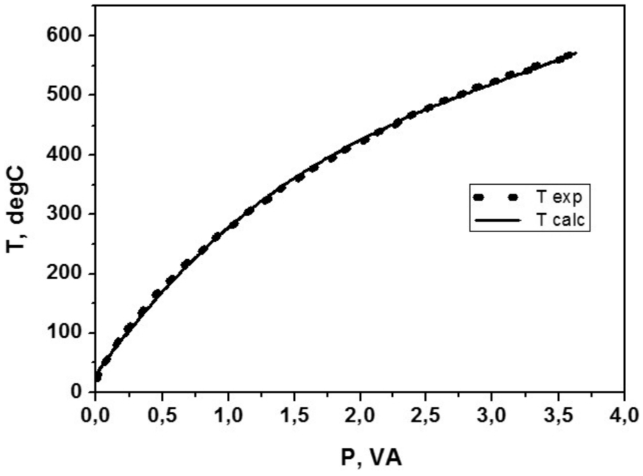

Fig. 5. The dependence of the heating temperature of the crystallized microwire on the applied thermal power. The curve plotted according to Eq. (1) with using experimental data is shown by dash symbols, and the solid line shows the graph plotted using the calibration characteristic (2).

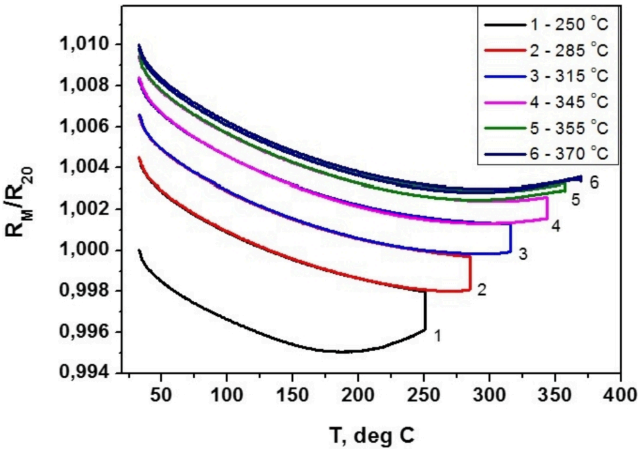

Fig. 6. Temperature dependences of the normalized resistance of the same microwire after several successive heating-holding-cooling cycles.

min was in the heating-holding-cooling mode (20 min each) at different values of the limiting heating power.

The above examples allow one to determine the characteristic features of the dependence of the resistance of microwires with the composition $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ when heated below the temperature of the onset of crystallization. These features include:

- heating up to each new set temperature is irreversible - the graph of each next heating coincides with the graph of the previous cooling;
- with each subsequent heating to a higher temperature, additional changes in the resistivity occur in the AFM;
- the maximum value of the resistance change is observed during heating in the temperature range from 230 °C to 320 °C;
- the first minimum in the temperature dependence of resistance is observed at a temperature of ~ 180 °C;
- with subsequent heating, the minimum of the temperature dependence of the resistance shifts towards higher temperatures up to ~ 280 °C when heating is above 320 °C.

The proposed technique for monitoring the resistance of a microwire can be used for controlling the temperature regime of heating a microwire sample of a certain series under the given measurement conditions. In the event of a change in measurement conditions and when testing samples of other compositions, it is necessary to determine a new calibration curve *T(P)*, which would allow determining the heating temperature under different measurement conditions. If necessary, preliminary results can be refined using more accurate methods of measurements.

4. Conclusion

The results of comparative studies of the properties of amorphous ferromagnetic microwires during heat treatment in a conventional furnace and by the Joule heating method are presented. It is shown that a microwire with the composition $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ undergoes a two-stage crystallization process when heated to 600 °C (4.0 W with Joule heating). Fully crystallized microwire dramatically changes its electrically conductive properties. The resistance of the microwire drops by 13% with respect to the initial value, while its temperature dependence of resistance $R_M(T)$ becomes linear with a characteristic TCR $\alpha = 315*10^{-6}$ 1/ °C and does not change with subsequent heating and cooling cycles.

This feature allows the use of crystallized microwires as resistance thermometers after their calibration. In addition, the graphs of the temperature versus power of Joule heating of such microwires can be used as a calibration characteristic to estimate the temperature of other samples of microwires of the same series, which are in an amorphous or partially crystallized state during Joule heating. The proposed method for recalculating the impacting thermal power into the appropriate temperature makes it possible to compare the resistive, magnetic, and structural-phase properties of microwires.

Our approach can be used for measuring the temperature of crystallized microwires under Joule heating for determining the heat-conducting properties of highly filled polymer composites with increased heat-conducting and strength characteristics. Due to the insulating glass sheath, microwires can be used to measure temperature in electrically conductive composite materials. Another promising area of applications of resistance thermometers based on crystallized microwires can be temperature control in the working volume of magnetic sensors based on the GMI effect and the corresponding control of temperature errors.

CRediT authorship contribution statement

S.A. Gudoshnikov: Conceptualization, Methodology. **V.I. Odintsov:** Investigation, Writing - review & editing. **B.Ya. Liubimov:** Methodology, Formal analysis. **S.A. Menshov:** Investigation. **M.N. Churukanova:** Investigation, Writing - original draft. **S.D. Kaloshkin:** Project administration, Funding acquisition. **G.N. Elmanov:** Conceptualization, Formal analysis, Methodology, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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