ISSN 1028-334X, Doklady Earth Sciences, 2006, Vol. 406, No. 1, pp. 46–51. © Pleiades Publishing, Inc., 2006.
Original Russian Text © Yu.G. Astrakhantsev, A.G. Lavrent'ev, V.E. Shcherbinin, G.S. Korzunin, V.L. Nekhoroshkov, A.P. Potapov, T.A. Sherendo, 2006, published in Doklady Akademii Nauk, 2006, Vol. 406, No. 1, pp. 89–94.

GEOPHYSICS

Prospects of the Application of Modern Soft-Magnetic Materials in Magnetometric Geophysical Equipment

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Received June 27, 2005

DOI: 10.1134/S1028334X06010120

In the last decade, the development of borehole magnetometry has been determined by the increasing demand for magnetometric equipment and by the broadening scope of its application. At present, two types of three-component digital ferroprobe borehole magnetometer-inclinometers have been developed at the Institute of Geophysics, Ural Division of the Russian Academy of Sciences. The first type is based on the second harmonic [1, 2], while the second one is of the time-pulse type [3]. This equipment has been widely used for the prospecting and exploration of iron, nonferrous and precious metals, and diamond and hydrocarbon deposits, as well as being utilized for the solution of technological problems in oil-and-gas fields [4]. The application of these devices in fundamental research into the oceanic and continental crust, including investigations into deep and superdeep boreholes [5] drilled in different regions of the former Soviet Union and abroad (Germany, Finland, and the Atlantic and Pacific oceans), became a new stage in the development of borehole magnetometry. In order to study the time variations in the Earth's magnetic field at geophysical observatories, magnetovariation stations, developed on the basis of the borehole ferroprobe magnetometer, are used in reference parametric boreholes and at the Kola Superdeep Borehole (Geolaboratory). In the course of geomagnetic investigations into such complex objects, specific features of measurements require not only highly sensitive, but also compact and reliable devices that can operate stably for long durations in the monitoring regime at high temperatures.

One of the pathways in the development of magnetometric equipment is the use of modern soft-magnetic materials and new technologies of their processing for manufacturing primary transformers of ferroprobe magnetometers. In recent years, researchers from the Institute of Geophysics and the Institute of Metal Physics have successfully carried out joint investigations into the application of amorphous and nanocrystalline materials with specified functional characteristics as ferroprobe cores and the identification of factors that can influence the operation of ferroprobes. According to the results of previous investigations [6], we chose a nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and Co-based amorphous (metalloid-free) alloys for further work. Their parameters and magnetic properties (in terms of temperature and time stability) suit the necessary conditions of the problem. In this work, we present the results of investigations into the abovementioned nanocrystalline alloy and metalloid-free alloy $Co_{81.5}Mo_{9.5}Zr_9$. The cores of ferroprobes were made in the form of stripes (length 15 and 32 mm, width 0.3 mm, and thickness 20 μm). The reference samples for controlling magnetic properties were also made in the form of stripes 100 mm long, 10 mm wide, and 20 μm thick. The cores and reference samples underwent all technological treatments under the same conditions. Amorphous stripes of the nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and metalloid-free alloy $Co_{81.5}Mo_{9.5}Zr_9$ (thickness 20 μm) were obtained by quenching the melt over a rotating disk. In order to obtain a nanocrystalline structure, the cores and reference samples of the first alloy were annealed in vacuum at 540°C for 1 h [7]. Thermomagnetic treatments (TMT) were carried out in the presence of constant ($TMT_=$), alternating $TMT_\sim$ ($f = 50$ Hz), and rotating [8] magnetic fields with heating up to 540°C, i.e., combining the thermomagnetic treatment with the phase transition of the alloy from an amorphous to nanocrystalline state. The Co-based amorphous alloy $Co_{81.5}Mo_{9.5}Zr_9$ was subjected to a similar treatment but in another temperature regime (annealing temperature 350°C). The changes in magnetic properties after technological treatments were checked on stripe samples by recording hysteresis static loops and the electromotive force (emf) of the Barkhausen jump fluxes (BJ) ε [9]. The BJ flux was visually observed on the oscillograph

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screen. The investigation of the phase composition of the magnetic component of alloy and the thermal stability of its magnetic properties in the operation temperature range of borehole magnetometric equipment was carried out using the method of thermomagnetic analysis by measuring the temperature dependence of normalized magnetic conductivity $\frac{\mu}{\mu_0} = f(T)$ of reference samples.

The influence of the treatment conditions of nanocrystalline alloy cores on the operation of ferroprobe sensors based on the principle of measuring the second harmonic was studied using a setup for investigating the characteristics of primary transformers [10]. The variations in the amplitude of the output signal A_2 of ferroprobe depending on the value of perturbation field H_m were recorded and internal noises of primary transformers were also studied. In addition, we studied the forms of the output signal of the time-pulse magnetometer depending on the type of treatment of nanocrystalline and amorphous alloy cores.

The table presents results of the computer processing of natural noises of ferroprobes with cores of nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ after $TMT_=$ and $TMT_\sim$ and after thermal annealing (TA) of this alloy at 540°C. For comparison, the table also shows similar data on the amorphous alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$, which is widely used in ferroprobe magnetometers, recorded after its annealing to remove quenching strains and the values of the BJ ε flux averaged over the emf period for three nanocrystalline samples that underwent the same treatments.

Analysis of the obtained results shows that the minimal value of the ferroprobe saturation field (the ferroprobe operates in the saturation regime) determined from the maximal value of the output signal of the ferroprobe $A_{2\max}$ corresponds to the excitation field of $H_m \sim 0.90$ A/cm (for the core that underwent $TMT_=$) and ~0.95 A/cm (for the core after $TMT_\sim$). Earlier, we found [10] that these treatments were followed by the highest values of magnetic conductivity at minimal values of the coercive force. We note for comparison that the core of the alloy subjected to thermal annealing has $H_m \sim$ 1.17 A/cm, while the core of amorphous alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ subjected to annealing has $H_m \sim$ 1.03 A/cm.

Magnetometric investigations in boreholes are carried out at temperatures critical for the operation of transistor devices (120°C or higher). The generators for excitation of the primary transformers of borehole magnetometer-inclinometers are located in the casing of the instrument. Therefore, for an increased reliability of the operation of such devices under industrial conditions, it is expedient to use ferroprobes with the minimal excitation field H_m.

Figure 1 shows results of laboratory measurements of natural noises of ferroprobes with cores of nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ subjected to TMT and TA treatments. The natural noise level of ferroprobes, which influences one of the main metrological characteristics of the instrument (sensitivity threshold), is a major operation factor of primary transformers of borehole magnetometers. This is a pressing issue in the geophysical investigation of superdeep boreholes and in the measurements of variations in the Earth's magnetic field recorded in geophysical observatories. The table presents results of the computer processing of measured natural noises of ferroprobes after various technological treatments of their cores as root-mean-square (rms) deviations for the 20-min-long interval of measurements. The results presented here suggest that the lowest level of this parameter corresponds to thermal treatment without the magnetic field. The minimal values of ε also support this conclusion.

Table

Alloy	Type of treatment	MSWD, nT	ε, mV	$H_{A2\max}$, A/cm
$Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$	TA	1.63	–	1.03
$Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$	TA	3.27	10.3	1.17
"	$TMT_=$	23.6	71	0.90
"	$TMT_\sim$	15.4	46	0.95

These data can be explained on the basis of an analysis of oscillograms for the envelope curves of BJ flux amplitudes for the nanocrystalline alloy studied here (Fig. 2). The distribution of BJ by the values of start critical fields was measured using a lay-on transformer [9] that records the normal component of the scattering field of the tested sample. Two regions of start fields observed in the oscillogram for the sample after $TMT_=$ treatment indicate the presence of two magnetic phases with different coercive forces. It is likely that these are the remains of the initial amorphous phase and the newly formed nanocrystalline phase α-Fe–Si. The method of thermal magnetic analysis showed the presence of two magnetic phases with different Curie temperatures $T_{c1} \sim 380$°C and $T_{c2} \sim 560$°C (Fig. 3), in contrast to one magnetic phase with $T_c \sim 570$°C in a nanocrystalline sample treated in an alternating magnetic field. In this case, the greater degree of material homogeneity after $TMT_\sim$ compared to $TMT_=$ is also reflected in the envelope curve of the BJ flux: the start fields of two phases become even, and the intervals of the start fields become narrower. The results obtained suggest that the degree of nanocrystalline alloy homogeneity plays a crucial role in the level of natural noises: the phase and magnetic homogeneity of the core material have an inverse correlation with the noise level of the ferroprobe.

Thus, the results obtained on the basis of the rms deviation ratio, which characterizes the natural noises and excitation fields H_m of the ferroprobe that takes into

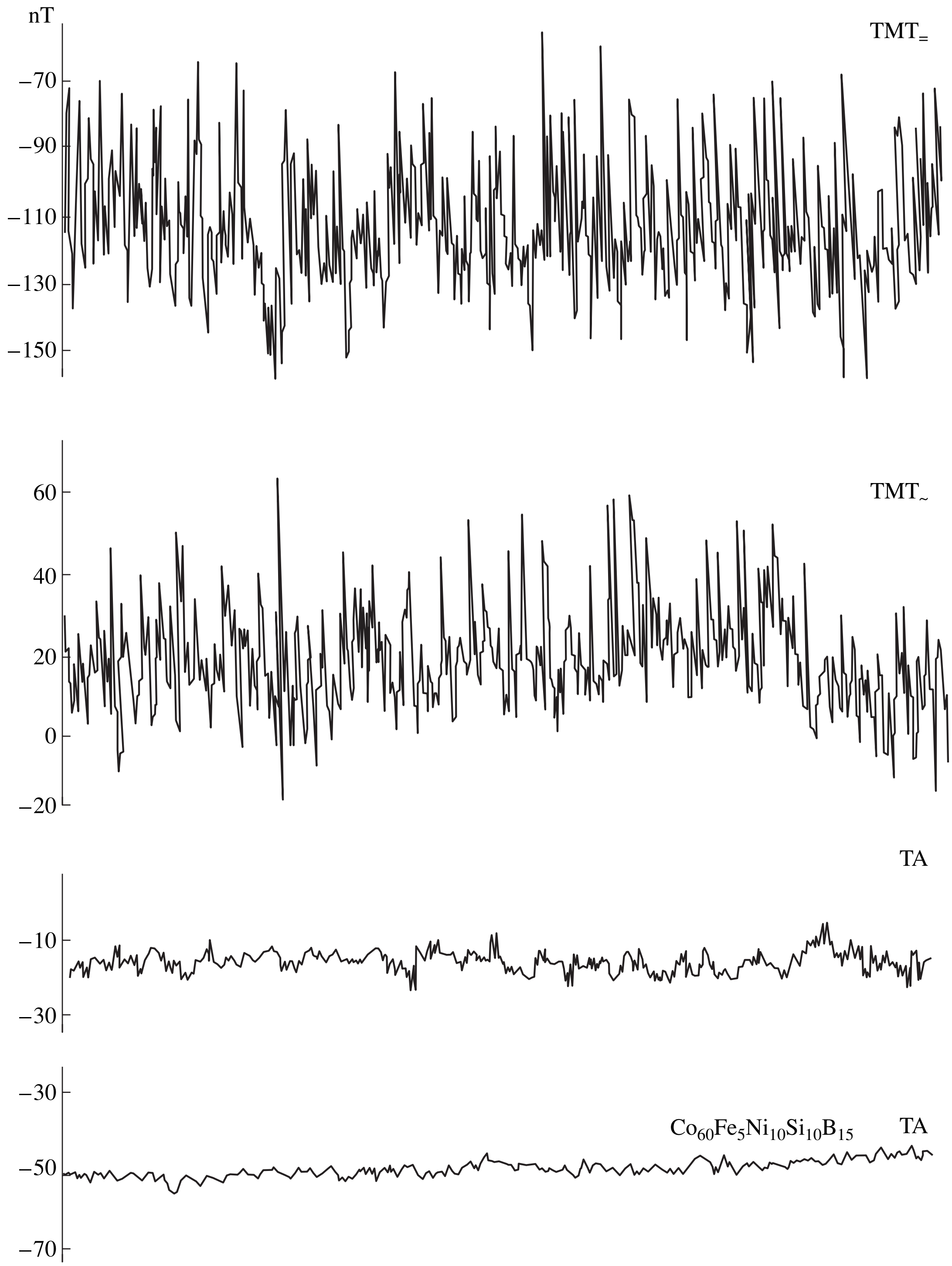

Fig. 1. Noises of ferroprobes with cores of nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ after different treatments as compared with amorphous alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ (record time 20 min).

account the amplitude of the output signal A_2, one can conclude that the annealing of nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ is effective for the cores of ferroprobes of a magnetometer operating on the second harmonic.

Figure 4 shows data on the shape of the output signal of the primary transformer of a time-pulse magnetometer operating at a frequency of 32 Hz as a function of the treatment conditions of the cores of ferroprobes made of nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and amorphous metalloid-free $Co_{81.5}Mo_{9.5}Zr_9$ alloys.

The method of time-pulse modulation with the application of one-element ferroprobes as primary transformers is used in this magnetometer. The princi-

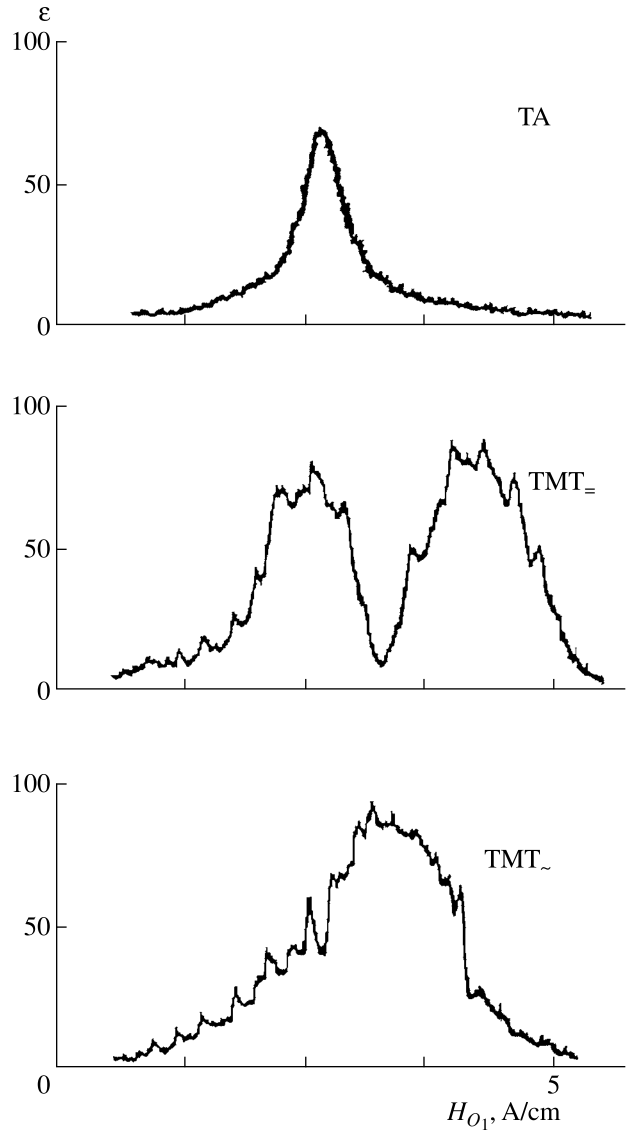

Fig. 2. Oscillograms of envelop curves of the amplitudes of Barkhausen ε jump fluxes in the alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ samples after different treatments.

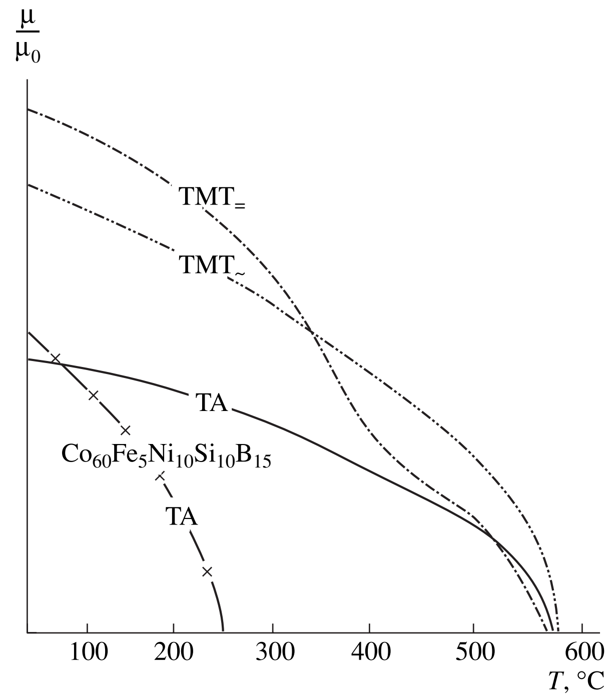

Fig. 3. Dependence of reduced magnetic conductivity $\frac{\mu}{\mu_0}$ on the temperature of nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and amorphous alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ after different technological treatments.

ple of magnetometer operation is based on the measurement of time intervals between pulses that appear after the magnetic reversal of the core of the ferroprobe by alternating magnetic field H_{exc}, which is induced in the excitation winding of the primary transformer. The measured external constant magnetic field H_{meas} biases the operation point so that the magnetic reversal of the core leads by time Δt, which is proportional to the measured field H_{meas}, during one half-period of the magnetizing alternating field and lags behind by the same time during the other half-period. The duration of the magnetic reversal front Δt_{fr} should not exceed 1 μs, because the duration of count pulses is equal to 1 μs when the comparator forms rectangular signals. The accuracy of the measurements of magnetic field decreases if the duration of the pulse front exceeds this value. This parameter of the ferroprobe is mainly determined by the material of the core. In our investigations, the standard was represented by the Co-based amorphous alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$, which has the smallest duration (*a*–*b*) of the pulse front Δt_{fr} (arrows in Fig. 4 show the moments of magnetic reversal of the core). This alloy has a significant disadvantage for its application in geophysical devices: it has a low Curie temperature (T_c = 250°C), which limits the scope of its application in a number of important objects, e.g., in superdeep boreholes and in specific conditions of oil and gas condensate fields. In addition, the thermal stability (Fig. 3) of magnetic properties of new alloys used in this investigation appeared to be significantly greater than that of the alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ taken as a standard. Therefore, we investigated nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and amorphous metalloid-free $Co_{81.5}Mo_{9.5}Zr_9$ alloys, which were selected in the course of preliminary investigations as being the most promising materials corresponding to the conditions of measurements and problems in magnetometric investigations of various geological objects [6]. These alloys have good magnetic parameters and, according to laboratory thermal magnetic investigations, a high thermal stability of magnetic properties in the operation temperature range of 0–300°C (Fig. 3). These properties are very important in geophysical research.

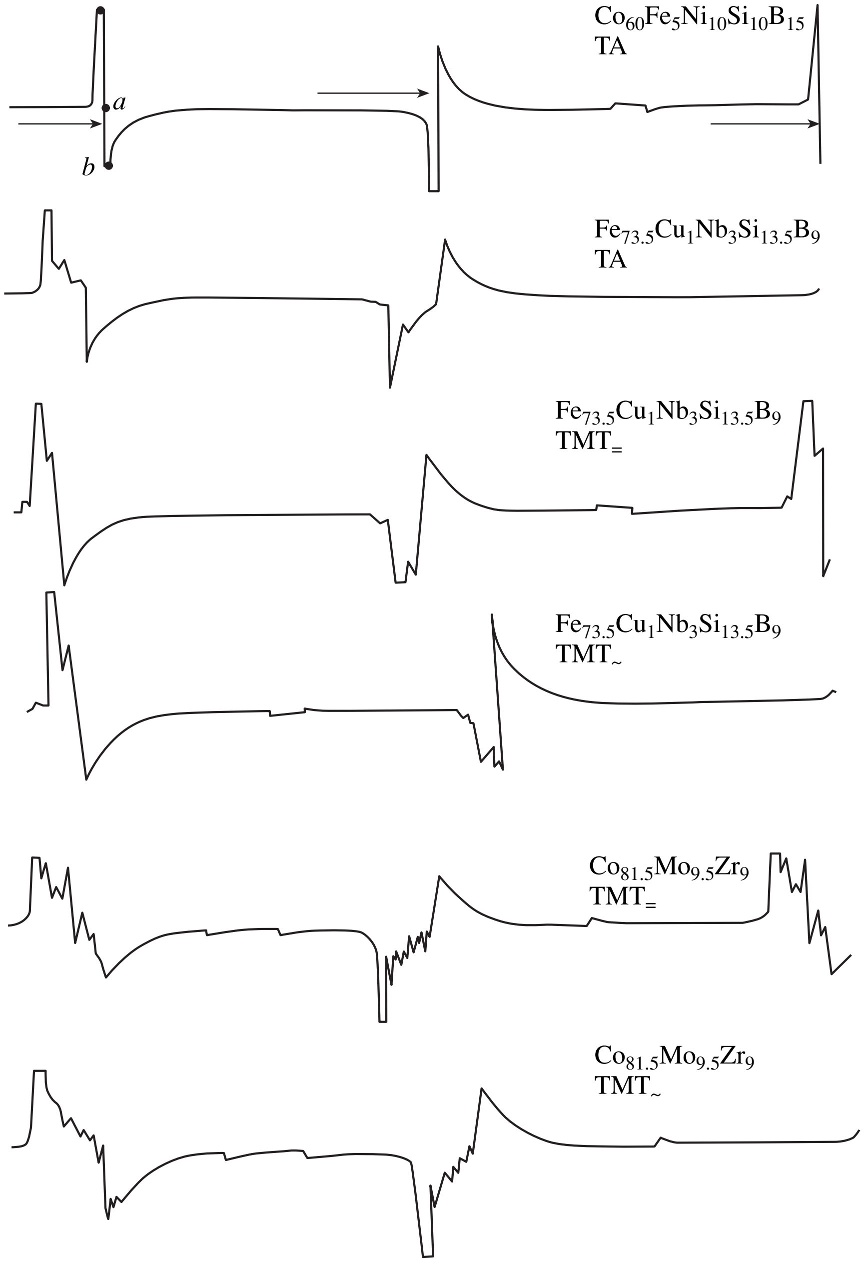

Fig. 4. Output signal oscillograms of the primary transformer of time-pulse magnetometer with cores of alloys $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and $Co_{81.5}Mo_{9.5}Zr_9$.

Cores made of the alloys were subjected to the TMT treatment in order to improve their magnetic parameters and to obtain impulse fronts of the output signal of the primary transformer, which correspond to the conditions of measurements. The shape of output signal of the primary transformer was investigated on a laboratory bench with the recording of oscillograms. Figure 4 shows results of the investigation with nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$. The oscillograms corresponding to different technological treatments (annealing, $TMT_=$, and $TMT_\sim$) of the cores of primary transformers indicate that the duration Δt_{fr} of magnetic reversal pulses is comparable with Δt_{fr} for alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ after the annealing. Thus, the accuracy of the measurements of geomagnetic field using nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ is comparable with the standard. In this method of geomagnetic field measurement, the range of its recording depends on the time of magnetic reversal of the core of the primary transformer. The time of magnetic reversal of the core (TA, $TMT_=$, and $TMT_\sim$) measured from the limiting hysteresis loop for alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ is greater than for alloy $Co_{60}Fe_5Ni_{10}Si_{10}B_{15}$ (TA), although treatment of the core in an alternating magnetic field ($TMT_{\sim}$) yields the best effect.

Similar investigations were carried out with the amorphous metalloid-free alloy $Co_{81.5}Mo_{9.5}Zr_9$. The results indicated that treatment of the core in an alternating magnetic field is more effective.

As a result, the influence of the phase composition, structural state, and degree of homogeneity of the magnetic structure of the material of ferroprobe cores on the functions of primary transformers and on the technical characteristics of borehole magnetometer-inclinometers was found (range of the measured geomagnetic field, threshold range of the instrument sensitivity, excitation regime, and the level of natural noises of the ferroprobe). It was shown that the phase composition and structural state of the investigated alloys, which are governed by the conditions of their production and the regime of technological treatment, can be purposefully designed according to the conditions of the application of measuring geophysical instruments.

The application of Co-based amorphous alloys and nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ after technological treatments for the purposeful improvement of their structural state and properties allows us to optimize the operation of primary transformers (ferroprobes) and widen the scope of the application of magnetometric devices, including high-temperature conditions of measurements in superdeep boreholes and geophysical monitoring.

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