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# Tunnel magnetoresistance of thin-film nanogranular metal–dielectric composites (*x*)Ni–(1−*x*)PNBZT

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**Keywords** ion-beam sputtering, metal–dielectric composites, Ni granules, percolation threshold, thin films, tunnel magnetoresistance

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Thin-film nanogranular metal–dielectric composites (*x*)Ni–(1−*x*)[$Pb_{0.81}Sr_{0.04}(Na_{0.5}Bi_{0.5})_{0.15}$][$(Zr_{0.575}Ti_{0.425})$]$O_3$ (denoted as (*x*)Ni–(1−*x*)PNBZT) were prepared by the ion-beam sputtering method. The X-ray diffraction analysis has revealed the presence of two phases in the composites: a crystalline Ni phase and an amorphous PNBZT phase. Composites (*x*)Ni–(1−*x*)PNBZT with compositions below the percolation threshold manifest a negative magnetoresistance, whose highest values of 0.13% at 287 K and 0.78% at 77 K are observed near the percolation threshold at $x_c = 35$ at.% for a composite sample of 0.31Ni–0.69PNBZT placed in a constant magnetic field with the strength of 8000 Oe. The magnetoresistance of the composites (*x*)Ni–(1−*x*)PNBZT is increased with the growth of the constant magnetic field strength, and the dependence remains the same as a polarity of the magnetic field is varied. The charge transport in the composites (*x*)Ni–(1−*x*)PNBZT over a temperature interval of 188–287 K is shown to be attributed to the inelastic resonant tunneling of electrons through energy barriers in a dielectric layer of PNBZT between crystalline ferromagnetic nanogranules of Ni. Therefore, to explain the experimentally established regularities of the negative magnetoresistance, the tunnel magnetoresistance model has been used.



**1 Introduction** Nanogranular composites of the metal–dielectric type, representing nanosized ferromagnetic granules randomly distributed in the bulk of a dielectric matrix, are very promising materials for practical applications [1]. The point is that such composites with compositions below the percolation threshold, thanks to the spin-dependent tunneling of electrons in them, have physical properties that are extremely important for condensed matter physics and applications, and greatly differ from physical properties of bulk metal–dielectric composites. For example, tunneling of electrons between ferromagnetic nanogranules in metal–dielectric composites with compositions below the percolation threshold placed in a magnetic field leads to the appearance of tunnel magnetoresistance [2]. For the first time, the tunnel magnetoresistance in metal–dielectric composites was found in the early seventies of the last century when Ni–$SiO_2$ composites were investigated [3] and now it has been studied in a vast number of nanocomposites both with one-element crystalline metallic phases (Co–$TiO_2$ [4], Co–$Al_2O_3$ [5], Co–ZnO [6], and so on), and with multiple-element amorphous metallic phases (FeCo–$SiO_2$ [7], $(Fe_{45}Co_{45}Zr_{10})_x(Al_2O_3)_{100-x}$ [8], $(Fe_xSn_{1-x})_{1-y}(SiO_2)_y$ [9], etc.). There are many theoretical models describing the tunnel magnetoresistance in metal–dielectric nanogranular systems, but only two of them are basic and have two different approaches to the description of the spin-dependent tunneling of polarized electrons. According to the first model proposed by Julliere [10, 11] and developed in works of Moodera [11, 12], a value of the magnetoresistance depends on the spin polarization of electrons tunneling through a dielectric barrier between metallic granules and the mutual direction of magnetic moments of granules. The second model, the Slonczewski model [13], developed by Inoue and Maekawa [14] also assumes that a value of the magnetoresistance depends on the spin polarization of electrons but the direction of electron spins is greatly related to the height of the tunnel barrier. This

model successfully agrees with the experimental data for the tunnel magnetoresistance in many metal–dielectric nanogranular systems.

Experimentally, many granular systems exhibit conductivity ($\sigma$) versus temperature dependences similar to that of $\sigma \propto T^{\gamma_n}$, where $\gamma_n$ is the exponent. Though this behavior has been observed for a set of composites, its origin is still controversial and various new special measurements have to be made to clarify it.

Until now the tunnel magnetoresistance has been studied in metal–dielectric composites, in which $SiO_n$ and $Al_2O_n$ were used as dielectric components. The choice of these dielectrics can be explained by their thermal stability and chemical inertness to metallic components over a wide temperature interval as well as by large values of the magnetoresistance in composites on their basis.

The present work seeks to establish basic regularities and the nature of the magnetoresistance in thin-film nanogranular composites of the form:

$$(x)\mathrm{Ni}-(1-x)[\mathrm{Pb}_{0.81}\mathrm{Sr}_{0.04}(\mathrm{Na}_{0.5}\mathrm{Bi}_{0.5})_{0.15}][(\mathrm{Zr}_{0.575}\mathrm{Ti}_{0.425})]\mathrm{O}_3$$

(hereafter $(x)$Ni–$(1-x)$PNBZT), where the amorphous ferroelectric PNBZT [15, 16] was used as a dielectric component. The choice of PNBZT was made because of its high phase transition temperature (320 °C) in the crystalline state and high piezoelectric coefficients (e.g., $d_{31} \approx 150$ pC/N). We chose the crystalline ferromagnetic Ni owing to its high phase transition temperature (358 °C) and a big value of the saturation magnetostriction ($\lambda_s \approx 50 \times 10^{-6}$) in low magnetic fields (<1 kOe). It means, that composites $(x)$Ni–$(1-x)$PNBZT can provide high magnetoelectric responses in low magnetic fields at temperatures above room temperature.

**2 Experimental** Nanogranular composites $(x)$Ni–$(1-x)$PNBZT were prepared by ion-beam sputtering of a target, consisting of a cast nickel base with nineteen ceramic plates of PNBZT on its surface, onto pyroceramic and silicon substrates with the thicknesses of 0.6 mm in the argon atmosphere at the pressure of $6.2 \times 10^{-4}$ Pa. The nickel base and ceramic plates of PNBZT had sizes of $280 \times 80 \times 10\,\mathrm{mm}^3$ and $80 \times 10 \times 2\,\mathrm{mm}^3$, respectively. The distance between PNBZT plates was increased from 0 mm for one edge of the nickel base to 16 mm for the other edge of the nickel base that enabled us to prepare composites with $x$ varying from 2 to 59 at.%. Composite samples were 10 mm × 3 mm × $d$ (μm) in sizes where the thickness ($d$) was varied over interval of 0.03–0.61 μm depending on the position of the target and the substrate relative to each other.

Samples on pyroceramic substrates were employed for measurements of the composition of composites and the dc electrical resistance ($R$) of composites depending on the composition, temperature, and the magnetic field, whereas samples on silicon substrates were employed for X-ray diffraction studies. The use of a silicon substrate instead of a pyroceramics one gives an opportunity to simplify the identification of X-ray diffraction patterns for composites owing to the decrease in a number of reflexes from a substrate.

The composition of composites was determined by electron probe X-ray microanalyzer JXA-840.

We measured $R$ of composites by the two-probe potentiometer method using a multipurpose voltmeter V7-78/1. Electrical contacts of beryllium bronze probes to a surface of every composite sample were ohmic and were realized through In-Ga eutectic contact pads. The distance between contacts was equal to 4 mm. Measurement error of $R$ was not more than 7%.

X-ray diffraction studies were carried out by means of a diffractometer Bruker D2 Phaser using $CuK_\alpha$ radiation.

To investigate surface morphologies of composite samples, a transmission electron microscope (TEM) Libra 120 was used.

The experimental study of the magnetoresistance of composites $(x)$Ni–$(1-x)$PNBZT consisted in the measurement of the dc electrical resistance of composites in the constant magnetic field.

We studied the longitudinal magnetoresistance in composites as the constant magnetic field was directed parallel to the direction of the current in a composite sample. The constant magnetic field with the strength $H_=$ varying from 24 to 8000 Oe was created by an electromagnet and was measured using a Hall sensor.

A value of the magnetoresistance was calculated by the following equation [17]:

$$\frac{\Delta R}{R(0)} = \frac{R(H_=) - R(0)}{R(0)} \cdot 100\%, \qquad (1)$$

where $R(H_=)$ and $R(0)$ are the dc electrical resistance of a composite in the presence and in the absence of the constant magnetic field, respectively.

**3 Results and discussion** Electron microscopic studies of composite samples in TEM have showed that composites $(x)$Ni–$(1-x)$PNBZT have a complex labyrinth-like structure, in which dark-colored Ni particles disperse in the light-colored PNBZT matrix (Fig. 1).

The average size of metallic granules in composites is some nanometers and takes on a value of about 2 nm for a composite with $x = 30$ at.% (Fig. 1). The average granule size $D$ was also estimated using an X-ray diffraction pattern for the composite 0.3Ni–0.7PNBZT (Fig. 2) by the Scherrer equation:

$$D = \frac{S\lambda}{B\cos\Theta}, \qquad (2)$$

where $S = 0.94$ is the Scherrer constant, $\lambda$ is the wavelength, $\Theta$ is the diffraction angle, and $B$ is the full width (in radians) of a diffraction peak at the half-maximum intensity [18].

Substituting $S = 0.94$, $B = 0.02$ radians, and $\Theta = 21.941°$ for a peak corresponding to Ni with the reflection plane (111) (Fig. 2) as well as $\lambda = 1.541$ Å ($CuK_\alpha$ radiation)

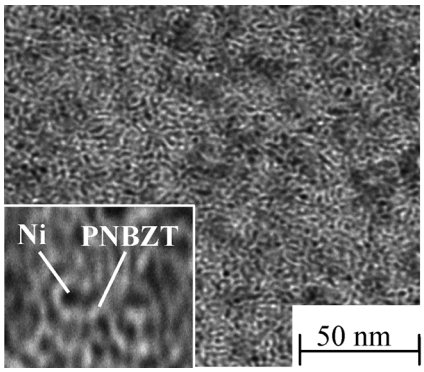


**Figure 1** Surface morphology of the 0.3Ni–0.7PNBZT thin-film heterostructure obtained by the TEM method. Insert shows a piece of the micrograph increased in eight times.

into Eq. (2), we obtain the average granule size of $D = 7.25$ nm, which by the order of magnitude coincides with $D$ determined by TEM (Fig. 1).

The X-ray diffraction analysis confirms the presence of two phases in composites $(x)$Ni–$(1-x)$PNBZT: the crystalline Ni phase and the amorphous PNBZT phase (Fig. 2). No intermediate phases were revealed. Crystalline Si reflexes in Fig. 2 correspond to a silicon substrate, which composites were sputtered on.

As the tunnel magnetoresistance is observed in nanogranulated metal–dielectric composites with compositions below the percolation threshold, it is important to know the position of the percolation threshold in our composites. For composites consisting of a dielectric matrix and conducting inclusions, the percolation threshold is most easily determined from the concentration dependence of the dc electrical resistance $R$ as at the critical concentration of inclusions an infinite conducting cluster with the law resistance is formed, that is easily observed in an experiment by the sharp decrease in the resistance of composites. Dependence $R(x)$ for composites $(x)$Ni–$(1-x)$PNBZT is demonstrated in Fig. 3.

Figure 3 shows the sharp decrease in $R$ with the increase in $x$ from 16 to 35 at.% testifying to the growth of a number

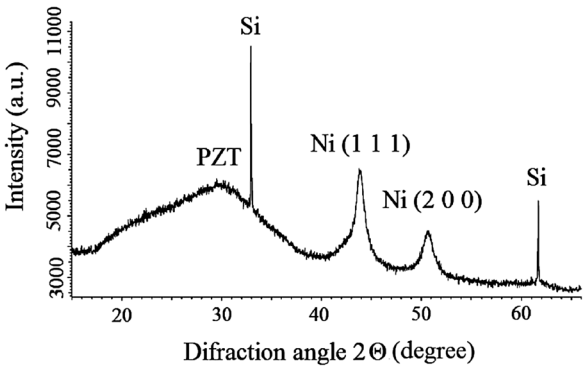


**Figure 2** X-ray diffraction pattern for the composite 0.3Ni–0.7PNBZT film on a monocrystalline silicon substrate with the orientation of (100) at room temperature.

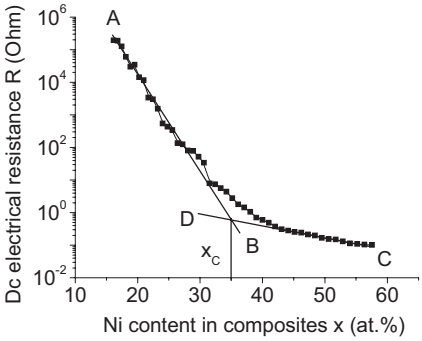


**Figure 3** Dependence of dc electrical resistance ($R$) with composition ($x$) for $(x)$Ni–$(1-x)$PNBZT composites at 287 K.

of individual Ni nanogranules in the PNBZT matrix, which join to form separate conductive clusters. In composites with such compositions, the transport of electric charges is mainly due to the conductivity of PNBZT, because it is essentially smaller than the conductivity of Ni, i.e., the electrical resistance of composites is decreased with temperature. The increase in $x$ within the limits of 35–57 at.% leads to the slight variation in $R$, since in this case separate clusters of Ni granules join together and form a continuous net of metal clusters, which provide, basically, the metallic-type conductivity. The percolation threshold $x_c$ for $(x)$Ni–$(1-x)$PNBZT is 35 at.% and was found in this work as a intersection point of straight lines AB and CD approximating a curve piece of maximal variation in the dc electrical resistance at 16 at.% $< x <$ 35 at.% and a piece, where the dc electrical resistance is slightly varied at 35 at.% $< x <$ 57 at.%, respectively (Fig. 3).

To find out a magnetoresistance mechanism in composites $(x)$Ni–$(1-x)$PNBZT with compositions below the percolation threshold, temperature dependences of the dc electrical conductivity ($\sigma(T)$) were studied. In work [19], we showed that several conductivity mechanisms are realized in composites over a temperature interval from 77 to 375 K: the Mott variable-range hopping conductivity [20], the inelastic resonant tunneling conductivity [21], the hopping conductivity on neighboring localized states [20], and the conductivity related to the thermally activated delocalization of charge carriers [20].

Over a temperature interval of 188–287 K dependences $\sigma(T)$ are well approximated by a power function [21]

$$\sigma_n = P\left(\frac{\Lambda^2}{\rho_0 c^5}\right)^{(n-1)/(n+1)} \frac{(ga^2n^2l)^n T^{\gamma_n} E^{\beta_n}}{al} \exp\left(\frac{-2l}{a(n+1)}\right), \quad (3)$$

where $a$ is the radius of a localized state, $l$ is the average distance between granules, $\gamma_n = n - 2/(n+1)$ is an exponent, $\beta_n = 2n/(n+1)$ is an exponent, $P$ is a coefficient, $\Lambda$ is a deformation potential constant, $\rho_0$ is the density of the matrix material, $c$ is the velocity of sound, $g$ is the density of

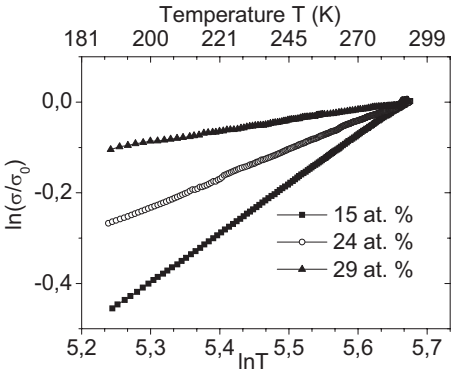


**Figure 4** Dependence of $\ln(\sigma/\sigma_0)$ versus $\ln T$ for $(x)$Ni–$(1-x)$ PNBZT composites for various $x$, where $\sigma_0$ is the dc conductivity at 287 K.

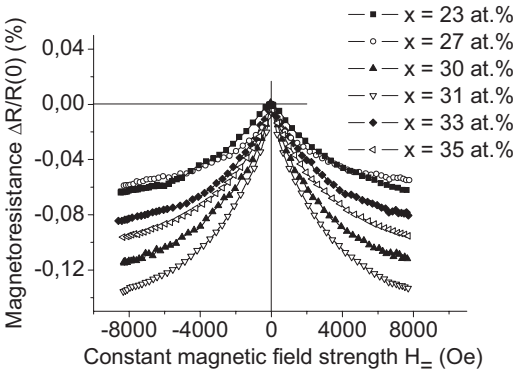


**Figure 5** Dependence of $\Delta R/R(0)$ with magnetic field strength ($H_=$) for $(x)$Ni–$(1-x)$PNBZT composites at 287 K.

localized states, and $E$ is the depth of a localized state in the barrier region, $n$ is the number of localized states.

A good approximation of the dependences $\sigma(T)$ is confirmed by straight lines in the coordinates $\ln(\sigma/\sigma_0)$ versus $\ln T$, with experimental dots well laying on the curves (Fig. 4). The values of $\gamma_n$ corresponding to slope angles of the lines for various composites are presented in Table 1.

The average conductivity between granules is the sum of conductivities over several channels

$$\sigma^{(gr)} = \sum_n \sigma_n. \tag{4}$$

The total conductivity $\sigma$ of a nanogranular structure below the percolation threshold is related to the tunnel conductivity between granules $\sigma^{(gr)}$. To the first approximation, it is usually assumed that the main contribution to $\sigma$ in Eq. (4) over a temperature interval $[T_n, T_{n+1}]$ is due to the first term, $\sigma_n$. In accordance with this approximation, $\sigma$ is a power function, which is determined by $\sigma^{(gr)}$ with $n = \langle n \rangle$ representing the number (averaged over the entire structure) of localized states in tunnel channels between granules. Using values of $\gamma_n$ (Table 1), we can calculate the average number of localized states between granules involved in electron transport at a given temperature [22]

$$\langle n \rangle = \frac{1}{2}\left[\gamma_n - 1 + (\gamma_n^2 + 2\gamma_n + 9)^{1/2}\right]. \tag{5}$$

**Table 1** Parameters of electron transport in $(x)$Ni–$(1-x)$PNBZT composites.

| $x$ (at.%) | $\gamma_n$ over a temperature interval of 188–287 K | $\langle n \rangle$ at 287 K |
|---|---|---|
| 15 | 1.07 | 1.79 |
| 24 | 0.62 | 1.44 |
| 29 | 0.24 | 1.16 |

Calculations showed that $\langle n \rangle$ decreases as the metal fraction in $(x)$Ni–$(1-x)$PNBZT composites is increased (Table 1). This agrees well with data obtained for other nanogranular metal–dielectric composites [22].

In works devoted to many granular metal–dielectric systems, the $\sigma \propto T^{\gamma_n}$ law is usually explained by the inelastic resonant tunneling of electrons through energy barriers in a dielectric layer between metallic nanogranules [21]. Therefore, the charge transport in our composites was supposed to be related to the inelastic resonant tunneling of electrons through barriers in a layer of PNBZT between Ni granules and the tunnel magnetoresistance model [14] was used for explanation of magnetoresistance regularities.

Measurements of the dc longitudinal magnetoresistance $\Delta R/R(0)$ were performed using samples of composites $(x)$Ni–$(1-x)$PNBZT with $x$ from 23 to 35 at.% placed in a constant magnetic field $H_=$ varying from 24 to 8000 Oe. Results are presented in Fig. 5.

One can see that the electrical resistance $R$ is decreased with $H_=$, i.e., the composites possess a negative magnetoresistance. The higher $H_=$ is applied, the higher the negative magnetoresistance is. A dependence of $\Delta R/R(0)$ on $H_=$ remains the same as the polarity of the magnetic field is varied. The highest value of the magnetoresistance at 287 K is 0.13% for the composite 0.31Ni–0.69 PNBZT at a constant magnetic field with the strength of 8000 Oe.

The magnetoresistance of the composites $(x)$Ni–$(1-x)$PNBZT depending on the content of Ni passes through a maximum for a composition with $x = 31$ at.% (Fig. 6), which is near the percolation threshold $x_c = 35$ at.%.

Features of the magnetoresistance of composites $(x)$Ni–$(1-x)$PNBZT obtained in our work can be explained as follows. As shown above, at 287 K in composites the charge transport takes place as a result of the inelastic resonant tunneling of electrons from one granule to another one through barriers in a dielectric layer, so the tunnel magnetoresistance model was used [14] for the explanation of a dependence of $\Delta R/R(0)$ on $H_=$. According to the model, the variation of $R$ of composites $(x)$Ni–$(1-x)$PNBZT with

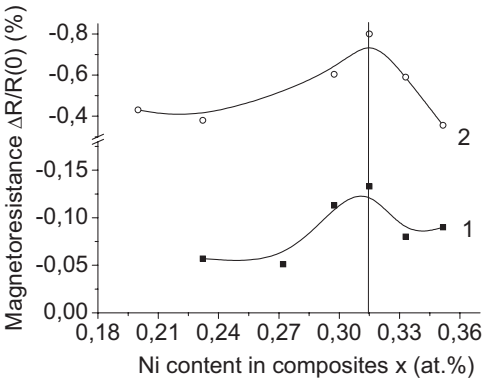


**Figure 6** Dependence of $\Delta R/R(0)$ with composition ($x$) for ($x$)Ni–(1−$x$)PNBZT composites in a constant magnetic field with $H_{=} = 8000$ Oe at: (1) 287 K and (2) 77 K.

compositions below the percolation threshold under the constant magnetic field is related to the spin-dependent tunneling of spin-polarized electrons through an amorphous dielectric PNBZT layer between neighboring ferromagnetic Ni nanogranules. In magnetized granules, the density of electronic states with oppositely directed spins are different and their energy electronic subbands are shifted on the energy scale because of the ferromagnetic ordering [22]. An energy subband of electrons, in which spins are co-directional with the magnetization vector of a granule, is completely filled. At the same time, a neighboring subband with electrons, whose spins are anti-directional with the magnetization vector of a granule, is partially filled. In this regard, densities of electronic states on the Fermi level in two subbands of a granule differ [22]. The probability of the electron tunneling between granules is maximal at identical directions of magnetization vectors of granules, as in this case electrons at the completely filled Fermi level in a partially filled subband of one granule can tunnel into vacant states in a partially filled subband with the same spin direction of a neighboring granule. The electron tunneling is the result of the identity of initial and finite energy states of electrons. If magnetization vectors of granules are oppositely directed, electrons on the Fermi level in a partially filled subband of a granule cannot tunnel into a neighboring granule, since for it a subband with the same spin direction is completely filled and vacant states on the Fermi level are absent [22]. Thus, the increase in $H_{=}$ leads to the alignment of magnetic moments of ferromagnetic granules along the field direction. The more $H_{=}$ is in value the bigger a number of ferromagnetic granules with magnetic moments along the field direction is. According to the model, this is equivalent to the growth of the tunneling current through a composite, to the decrease in the resistance of a composite and, as a consequence, to the increase in the negative magnetoresistance of a composite.

A maximum of $\Delta R/R(0)$ against $x$, shown in Fig. 6, could be explained in such a manner. The probability of electron tunneling depends on the thickness of a dielectric barrier between granules. At first, it is increased with $x$ in composites ($x$)Ni–(1−$x$)PNBZT because of the decrease in the distance between granules, becomes maximal at $x = 31$ at.% (for the minimal thickness of a barrier), and then decreases at $x > 31$ at.% as granules begin contacting with each other and in composites the change of a conductivity mechanism from the dielectric type to the metallic type occurs. Thus, a maximum is observed in the dependence of $\Delta R/R(0)$ on $x$.

As it appears from experimental studies [22], at some constant magnetic field the magnetization of nanogranular metal–dielectric composites is increased as temperature is lowered, that is apparently caused by the decrease in thermal fluctuations of magnetic moments of granules with regard to the field direction. Meanwhile, processes of magnetization reversal are responsible for the magnetoresistance of composites [5]. Consequently, one can expect the increase in the magnetoresistance of composites with the temperature reduction and the expectation is confirmed by an experiment (Fig. 7).

The comparison of experimental data in Figs. 5 and 7 indicates the significant growth of the magnetoresistance of composites at the decrease in temperature from 287 to 77 K (six times for composite 0.31Ni–0.69PNBZT in the constant magnetic field with the strength of 8000 Oe). Meantime, the course of the curve $\Delta R/R(0)$ on $H_{=}$ at 77 K is the same as at 287 K.

The growth of the magnetoresistance of composites at the decrease in temperature can be understood from the following considerations. Magnetoresistance of composites results from the directional effect of the constant magnetic field on magnetic moments of Ni granules. In the constant magnetic field, magnetic moments of granules are aligned in the direction of the field, and the number of aligned magnetic moments is increased with the field strength. However, the thermal energy $kT$ prevents the alignment of magnetic moments along the magnetic field direction decreasing the magnetization of composites. Hence, a value

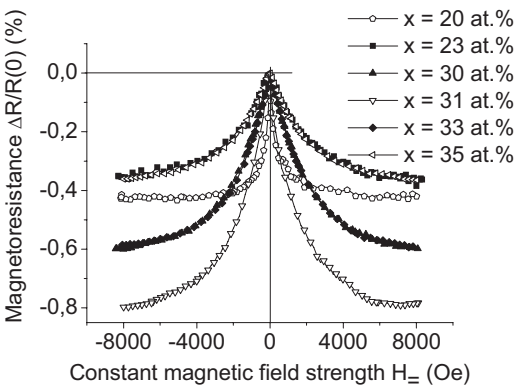


**Figure 7** Dependence of $\Delta R/R(0)$ with magnetic field strength ($H_{=}$) for ($x$)Ni–(1−$x$)PNBZT composites at 77 K.

of the magnetoresistance of composites at a certain magnetic field is the greater, the lower temperature is.

It should be pointed out, that for composites $(x)$Ni–$(1-x)$PNBZT the position of the peak in the dependence $\Delta R/R(0)$ on $x$ does not change when the temperature is lowered (Fig. 6).

The magnetoresistance values for the composites $(x)$Ni–$(1-x)$PNBZT are close to the respective values obtained for many nanogranular metal–dielectric composites [22]: $(Co_{41}Fe_{39}B_{20})_x(SiO_n)_{100-x}$, $(Co_{86}Nb_{12}Ta_2)_x(SiO_n)_{100-x}$, $(Co_{86}Nb_{12}Ta_2)_x(Al_2O_n)_{100-x}$, and so on.

**4 Conclusion** Using the ion-beam sputtering method, nanogranulated metal–dielectric composites $(x)$Ni–$(1-x)$PNBZT with $x$ from 2 to 59 at.% have been successfully prepared. The average size of ferromagnetic granules in composites is some nanometers and is of about 2 nm for the composite 0.3Ni–0.7PNBZT. Over a temperature interval of 188–287 K, the dc electrical conductivity of composites $(x)$Ni–$(1-x)$PNBZT obeys the $\sigma \propto T^{\gamma_n}$ law making it possible to relate the electrical conductivity of the composites to the inelastic resonant tunneling of electrons through a dielectric PNBZT layer between neighboring ferromagnetic nanogranules of Ni. This fact enabled us to use the tunnel magnetoresistance model for explaining the regularities of the magnetoresistance of composites depending on the constant magnetic field strength and the composition at 287 K. The composites manifest a negative magnetoresistance under constant magnetic field directed along the electric current and the value of the magnetoresistance is increased with the growth of the constant magnetic field strength. The composite 0.31Ni–0.69PNBZT having a composition close to the percolation threshold ($x_c = 35$ at.%) owns the highest value of the magnetoresistance of 0.13% at 287 K and 0.78% at 77 K in the constant magnetic field with strength of 8000 Oe. The concentration dependence of magnetoresistance for the composites $(x)$Ni–$(1-x)$PNBZT passes through a maximum corresponding to $x = 31$ at.%. The field and concentration dependences of magnetoresistance for the composites are well explained in terms of the tunnel magnetoresistance model.

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