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Relaxation of the dielectric response in thin films of vanadium dioxide

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Abstract. The results of studying the processes of dielectric relaxation in thin nanocrystalline films of vanadium dioxide are presented. The existence of a non-Debye relaxation process was revealed, which is due to the presence of a distribution of relaxators over relaxation times according to the Cole-Davidson model. The activation energy of the dielectric relaxation process was found to be $E_p = (0.9 \pm 0.1)$ eV. The observed regularities are explained by a model which views the system as a set of relaxators whose physical parameters have different numerical values due to the Gaussian size distribution of nanocrystallites of the VO_2 film. The temperature change in the parameters of the dielectric relaxation process, detected at T = 340 K, indicates that a complex Mott-Peierls semiconductor-metal phase transition occurs at a given temperature in the VO_2 nanocrystalline film.

Keywords: dielectric response, thin films, vanadium dioxide, distribution of relaxators, relaxation time distribution function.

Introduction

Vanadium dioxide (VO_2) occupies a special place among metal oxides, since vanadium is an element with an unfinished *d*-shell, and therefore it is characterized by the presence of strong correlation effects (Ilinskii et al. 2012). Relaxation processes in systems of this kind have a number of unusual properties, the physical nature of which is unclear. This ensures that interest in fundamental research on this material remains. In addition, thin-film oxidized VO_2-based structures are widely used in electronics and optoelectronics (Oleinik 2004). The operation of such devices as thermal relays, light flux restrictors and ultrafast systems for processing optical information is based on the semiconductor-metal phase transition, which occurs in nanocrystalline layers at a critical temperature T_c, providing a record-breaking short (femtosecond) response to external electromagnetic influence. The high sensitivity of nanocrystalline vanadium dioxide layers to external influences makes it convenient to control the temperature of the semiconductor-metal phase transition in a wide range of variation in the numerical values of its parameters and opens up the possibility of creating new generation optoelectronic systems based on VO_2.

In connection with the relevance of the search for solutions to the problems described above, the goal of this work was to use dielectric spectroscopy to reveal the features of dielectric relaxation processes in thin nanocrystalline vanadium dioxide layers and their relationship with the processes occurring in VO_2 nanocrystallites in the region of semiconductor-metal phase transition.

The advantage of the dielectric spectroscopy method is that this method allows one to monitor directly the change in the dielectric properties of a material in wide frequency and temperature ranges. The analysis of dielectric spectra makes it possible to reveal the dielectric and thermodynamic parameters of VO_2 nanocrystalline layers—such as the frequency spectra of the permittivity ε, relaxation times τ, the tangent of dielectric loss angle tgδ, the value of the activation energy E_a, and many other parameters that are inaccessible to study by other research methods (Castro et al. 2017; Zyryanova et al. 2017). The advantage of dielectric spectroscopy is, in addition, its high sensitivity to changes in the state of extremely thin nanocrystalline films (less than 100 nm thick) of strongly correlated materials, which opens up the possibility of high-precision express control of their electrophysical parameters.

Dielectric spectroscopy is a research method which easily provides reliable information on the parameters of the investigated dielectric thin-film materials in those frequently encountered situations when relaxation processes are not associated with the presence of one relaxation time, but rather with a set of relaxation times varying over a wide range. In such cases, dielectric spectroscopy solves the problem of constructing a function by distributing the relaxation times over their numerical values.

The relaxation time distribution function is the time dependence of the differential distribution function of the "time" density of relaxators dN/dτ—that is, the number of relaxators per unit time interval. The integral of this function with an infinite upper limit is equal to the total number of relaxators. The integral of this function with a variable upper limit is equal by definition to the integral distribution function of the number of relaxators over relaxation times. The presence of such a distribution may be due to the manifestation of physical processes of different nature in the process of relaxation screening of the external electric field.

In the case of dominance of purely electronic processes, it turns out that, following the application of an external electric field, it takes some time to establish a stationary distribution of internal fields and form a stationary concentration of free electrons in the space charge region. The two-parameter empirical Havriliak-Negami (HN) function can be used to obtain a general mathematical description of relaxation processes in thin films of strongly correlated materials (in particular, VO_2) and experimental dielectric spectra of such films (Schönhals, Kremer 2012). This makes it possible to establish the position (τ_0) of the maxima on the time scale and determine the parameters α and β of the HN function for relaxation processes described by formula:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\Delta\varepsilon}{\left[1+(i\omega\tau)^{\alpha_{HN}}\right]^{\beta_{HN}}}, \tag{1a}$$

where ε_∞ is the high-frequency limit of the real part of the permittivity, $\Delta\varepsilon$ is the dielectric increment (the difference between the low-frequency and high-frequency limits), $\omega = 2\pi f$, and α and β are the shape parameters describing, respectively, symmetric ($\beta = 1$, $0 < \alpha < 1$) Cole-Cole distribution and asymmetric ($0 < \beta < 1$, $0 < \alpha < 1$) distribution of relaxators according to their times.

The procedure for obtaining an analytical expression for the function $G(\tau)$ of the temporal distribution of relaxators over their times is reduced to solving the corresponding inverse problem, in which the expression for the function G(τ) is postulated in a simple form using several "free" parameters that are found from the relation (2):

$$\varepsilon^*(\omega) = \varepsilon_\infty + (\varepsilon_s - \varepsilon_\infty)\int_0^\infty \frac{G(\tau)}{1+i\omega\tau}\,d\tau\,, \tag{2}$$

where ε_s is the dielectric constant at extremely low frequencies, and τ is the integration parameter.

In this ideology, for the case of simple Debye relaxation with one single relaxator ($\tau = \tau_0$), the function G(τ) is a delta function. For a more complex relaxation, it is necessary to introduce free parameters into the function G(τ), which for the function G(τ) with parameters α, β, and τ_0 lead to an analytical expression for the simplest distribution function G(τ) of the form (Havriliak, Negami 1967):

$$G(\tau)=\frac{\left(\frac{\tau}{\tau_0}\right)^{\beta\alpha}\sin(\beta\Theta)}{\pi\tau(\left(\frac{\tau}{\tau_0}\right)^{2\alpha}+2\left(\frac{\tau}{\tau_0}\right)^{\alpha}\cos(\pi\alpha)+1)^{\beta/2}}\,, \tag{3}$$

where

$$\Theta=\arctan(\frac{\sin(\pi\alpha)}{\left(\frac{\tau}{\tau_0}\right)^{\alpha}+\cos(\pi\alpha)}) \tag{4}$$

and $0\le\Theta\le\pi$.

In this article, we only use the function G(τ) in its simplest form.

Experimental procedure

Thin-film structures based on vanadium dioxide nanocrystals were obtained by laser ablation by bombarding a V target with radiation from a pulsed neodymium-doped yttrium aluminum garnet IR laser. The radiation power density was 10^7–10^9 W/cm^2, and the radiation pulse duration was 20 ns. The temperature of the substrate during the synthesis was maintained in the range of 400–650°C. The oxygen pressure in the chamber varied within the range from 10^{-2} to $2 \cdot 10^{-1}$ mm Hg. This made it possible to synthesize nanocrystalline layers of vanadium dioxide with nanocrystallites of different sizes. The morphology of the VO_2 film was controlled by analyzing the atomic force images of the film (Il'inskii et al. 2018). The surface area of the film samples was 10×10 mm^2, the thickness of the films was 900 Å, and the resistivity was 7×10^6 Ohm^{-1} m^{-1} at room temperature. The resulting VO_2 films consist of one layer of adjacent columnar nanocrystallites which are in contact with each other by their lateral faces (Fig. 1). Fig. 1 shows a side cut of a monolayer of VO_2 crystallites in the form of a step obtained by the etching method.

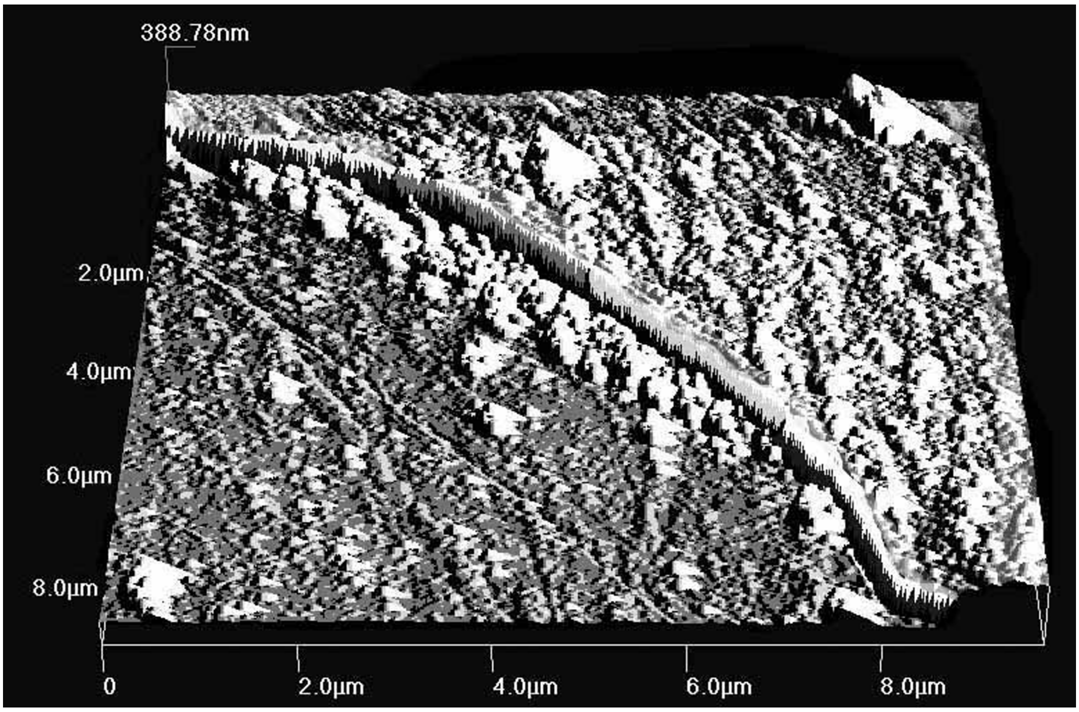

Fig. 1. Atomic-force image of the film surface and its end, obtained by local etching of the film

The measurements of the dielectric spectra of the layers under study were carried out on a Concept-81 spectrometer (Novocontrol Technologies GmbH) designed to study the dielectric and electrical conductivity properties of a wide class of materials. The measurements were carried out in the frequency range $f = 10^2$ Hz…10^6 Hz and temperatures T = 273 K…373 K.

The complex permittivity spectra were calculated from the impedance spectra using the following formulas:

$$\varepsilon^* = \varepsilon' - i\varepsilon'' = \frac{-i}{\omega Z^*(\omega)} \frac{1}{C_0}, \tag{5}$$

where $C_0 = \frac{\varepsilon_0 S}{d}$ is empty cell capacity.

Experimental results

The frequency dependence of the real part of the permittivity ε' at different temperatures for vanadium dioxide films is shown in Fig. 2. The figure shows that ε' decreases with increasing frequency, taking values from 13.7 to 11.6 for a temperature of 273 K. With an increase in temperature, this decrease persists, and the point of the maximum slope of the drop shifts towards high frequencies. For temperatures above T = 340 K, no drop is observed in the frequency range accessible for registration.

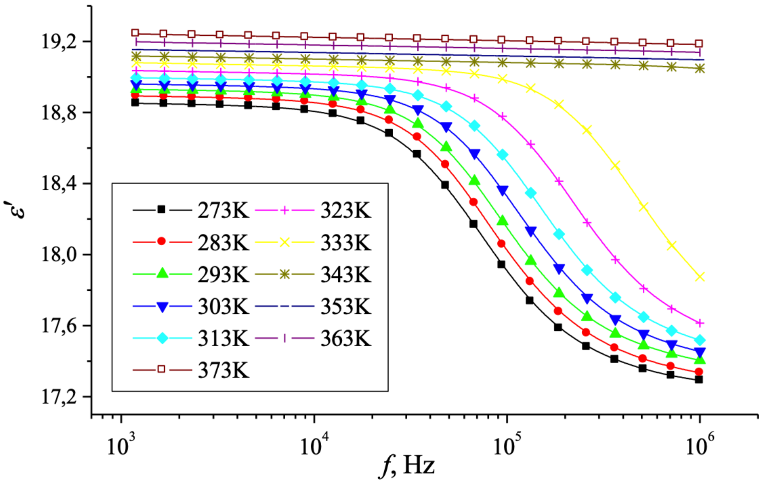

Fig. 2. Frequency dependence of the permittivity ε' at different temperatures

Fig. 3 shows the temperature dependence of the permittivity ε' for a frequency of 10^6 Hz, which follows from the data in Fig. 2. The figure shows that ε' increases with increasing temperature, experiencing a jump in the temperature range T = 340 K.

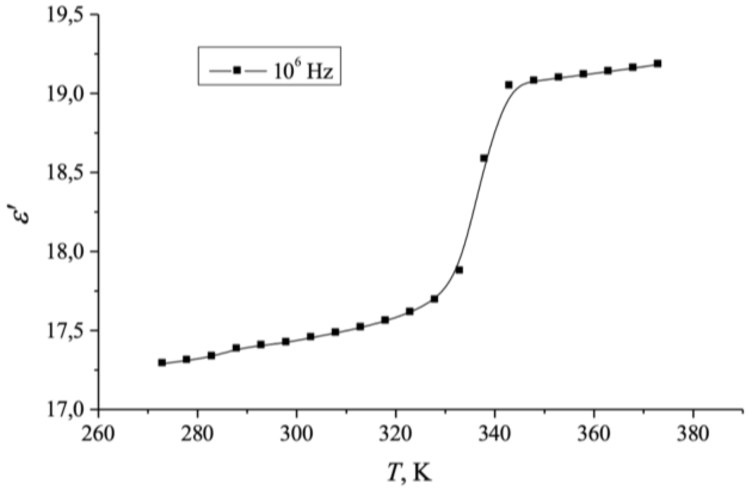

Fig. 3. Temperature dependence of the permittivity ε' of thin nanocrystalline vanadium dioxide layers

Fig. 4 demonstrates that with increasing temperature, the maximum of the frequency dependence of ε'' shifts towards higher frequencies.

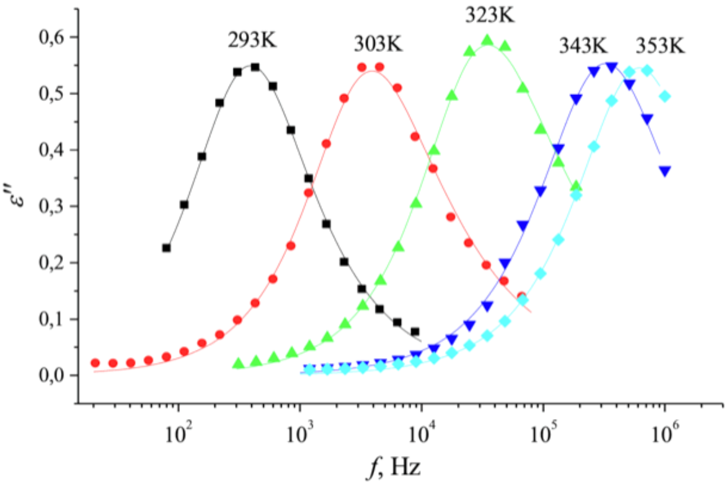

Fig. 4. Frequency dependence of the loss factor ε'' at different temperatures

From the data shown in Fig. 3 and Fig. 4, the frequency dependence of the dielectric loss tangent can be obtained according to the expression $tg(\delta) = \varepsilon''/\varepsilon'$—this expression is not shown, since it behaves similarly to ε'' with changes in frequency and temperature.

Fig. 5 for T = 303 K shows the Cole-Cole diagram (dependence $\varepsilon''(\varepsilon')$) which has the form of a slightly deformed semicircle—this form is due to the asymmetric distribution of the density of relaxators over the relaxation times. Note that at other temperatures in the range of 273–373 K the shape of the diagram remains practically unchanged, and, therefore, no diagrams for other temperatures are shown in Fig. 5.

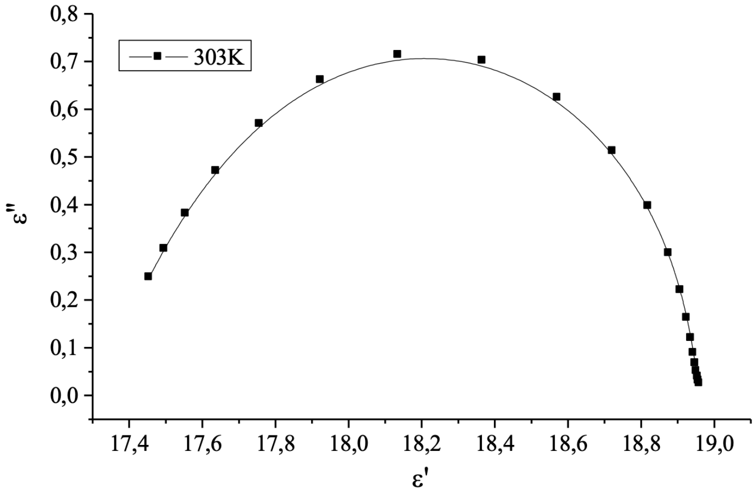

Fig. 5. Cole-Cole diagram of VO_2 layers at temperature T = 293 K (at temperatures T = 323 K, T = 343 K and T = 353 K, the shape of the diagram remains practically unchanged)

The form of the relaxation time distribution function for vanadium dioxide layers is shown in Figs. 6–7 for a set of temperatures: 293, 323, 343 and 353 K. We point out that the integral of this function within the limits (0–∞) is equal to the total number of relaxators. The integral of this function with a variable upper limit is equal by definition to the integral distribution function of the number of relaxators over relaxation times. Fig. 7 shows that at high temperatures $T > T_c$—i.e., at T = 353 K — this function has a symmetrical form. At T = 343 K (that is, near the semiconductor-metal phase transition) the function exhibits a slight asymmetry. At low temperatures, far from the semiconductor-metal phase transition, the time distribution functions are sharply asymmetric (Fig. 6). In addition, Figs. 6–7 show that, as the temperature rises, the area given by the integral of the above function increases, and it is especially strong in the temperature range of the semiconductor-metal phase transition—that is, the total number of relaxators grows.

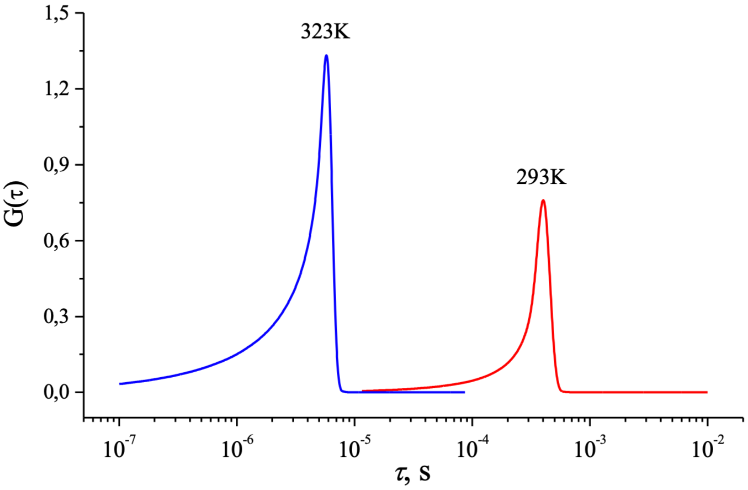

Fig. 6. Form of the distribution function of relaxation times for vanadium dioxide layers at low temperatures up to semiconductor-metal phase transition. The width of the peak of the distribution function at T = 293 K is $\Delta\tau = 2 \cdot 10^{-4}$ s ($\alpha = 0.99 \pm 0.03$; $\beta = 0.88 \pm 0.03$), at T = 323 K it is $3 \cdot 10^{-6}$ s ($\alpha = 0.98 \pm 0.03$; $\beta = 0.62 \pm 0.03$)

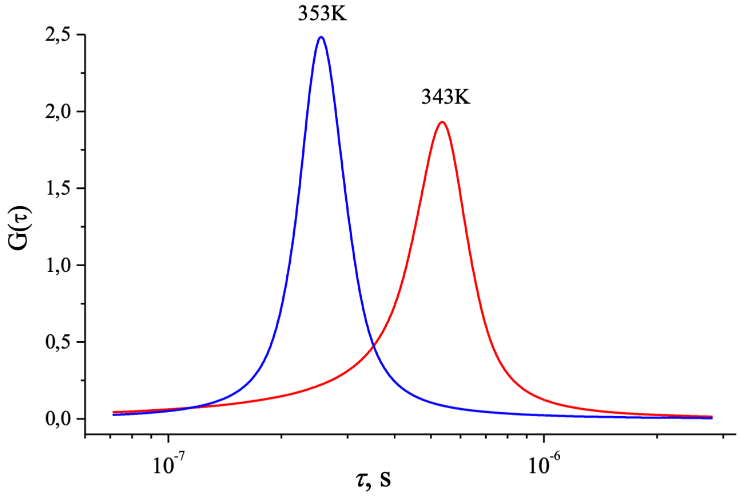

Fig. 7. Form of the distribution function of relaxation times for vanadium dioxide layers at high temperatures after the phase transition. The width of the peak of the distribution function at T = 353 K is $\Delta\tau = 2 \cdot 10^{-7}$ s ($\alpha = 0.96 \pm 0.03$; $\beta = 1.00$), at T = 343 K it is $4 \cdot 10^{-7}$ s ($\alpha = 0.98 \pm 0.03$; $\beta = 0.87 \pm 0.03$)

The temperature dependence of the frequency at which the maximum loss $\varepsilon''_m(\tau_0)$ is observed makes it possible to determine the activation energy of relaxation processes—i.e., the energy barrier required to excite relaxators. The activation energy calculated in Arrhenius coordinates from the slope of the temperature dependence of the most probable relaxation time turned out to be $E_a = (0.9 \pm 0.1)$ eV (Fig. 8).

The discussion of the results

Fig. 2 shows that the real part of the dielectric permittivity has decreased from the value ε_s (13.6) by the value Δε (13.6 – 11.7 = 1.9) in accordance with formula (2). The monotonic behavior of the frequency dependence ε(f) is determined by the function G(τ)—that is, by the presence of certain type of relaxators. We believe that the nature of the relaxators is determined by the charge-discharge processes of the capacitances C_i of the film elements and the corresponding sections C_{is} of the substrate, charged through the resistances R_i, which are the resistances of the nanocrystallites of the film at a given temperature. The corresponding equivalent circuit contains C_i and R_i connected in parallel, to which C_{is} is connected in series. In this case, R_i, as well as the conductivity of film nanocrystallites, depend on the temperature T (Ilinskii et al. 2020), while the capacitance values do not depend on T.

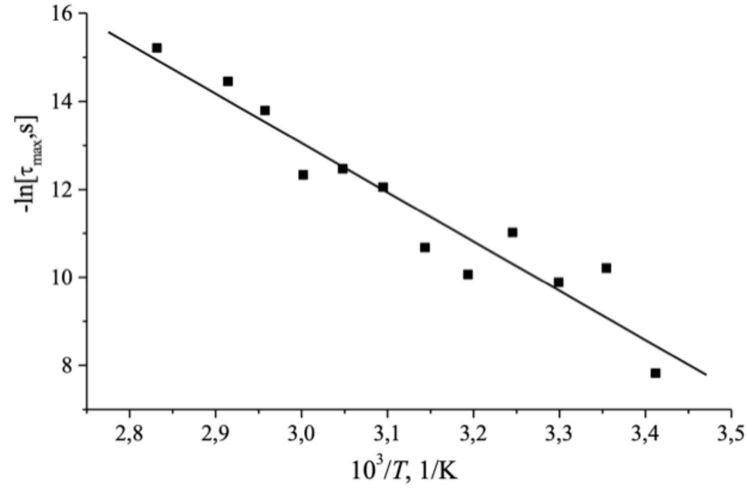

Fig. 8. Temperature dependence of the most probable relaxation time in thin VO_2 layers

The shape of the decay of the function ε(f) is well described by formula (2), in which the function G(τ) is a function of the parameters of one type of relaxators with a characteristic distribution of their “time” density over relaxation times. Of course, the presence of one type of relaxators does not mean that there is one single relaxator (in this case, we would have the function G(τ) in the form of δ-function). We are talking about the presence of relaxators of the same nature with a certain distribution of their relaxation times. That is, the Debye approximation, characterized by a relaxator with a single frequency, is not realized in this case. This conclusion is also confirmed by the shape of the Cole-Cole diagram $\varepsilon''(\varepsilon')$: the presence of one semicircle indicates relaxators of the same nature, and its asymmetry indicates that the Debye approximation is not realized. The form of this function for temperatures of 293–313 K is shown in Fig. 6.

The values of properties exhibited by this type of relaxers are widely dispersed, which is, in our opinion, caused by the similarly wide dispersion of the sizes and size-related electrophysical properties of nanocrystallites (Ilinskii, Castro, Pashkevich et al. 2020).

Figs. 2 and 6–7 show that changes in the characteristic features of the function ε' and the value τ_0 are shifted towards high frequencies up to 10^7 Hz (short times). We associate such a shift with a thermal drop in the resistance R_i of nanocrystallites and, accordingly, a decrease in τ_0 from $3 \cdot 10^{-4}$ s for T = 293 K to $5 \cdot 10^{-7}$ s for T = 343 K. This is confirmed by the behavior of the frequency dependence of the loss factor ε” at different temperatures (Fig. 3). Since the concentration of free electrons increases, and the resistance of nanocrystallites decreases, with increasing temperature, the maximum of dielectric losses ε'' shifts to the high-frequency region. The tangent of the dielectric loss angle $tg(\delta) = \varepsilon''/\varepsilon'$ behaves in a similar way. The monotonic shift of the position of the $tg\delta$ maximum toward high frequencies with increasing temperature up to 335 K also indicates a monotonic decrease in the electrical resistance caused by a thermal increase in the concentration of free electrons in the conduction band. We point out that at T = 340 K there is a sharp increase in the frequency at which the maximum $tg\delta$ (*f*) is located. We attribute this increase to the completion at this temperature of the semiconductor-metal phase transition at Tc = 340 K. Continuous metallization of nanocrystallites at $T > T_c$ forces one to switch to a fundamentally different equivalent electrical scheme describing relaxation processes. In this case, in the equivalent circuit of the sample, only the capacity of the substrate remains, which we consider to be practically ideal and which is charged with electrons through the mass of film nanocrystallites metallized at $T > T_c$.

The asymmetry of the peaks (Figs. 6, 7) is associated, in our opinion, primarily with the size distribution of nanocrystallites in the film, which at each temperature gives the distribution of nanocrystallites over the concentration of free electrons—that is, over the screening times of the external electric field by a gas of free electrons. In addition, interactions between electrons, which are characteristic of the highly correlated VO_2 material, contribute to the asymmetry.

Notably, at high temperatures (T = 353 K—i.e., at $T > T_c$), the relaxation time distribution function G(τ) has a symmetric form, and its half-width is $\Delta\tau = 10^{-7}$ s. If this function is described by the HN function, then for it α = 0.96 and β = 1. In the temperature range near the semiconductor-metal phase transition (T = 343) K, there is some asymmetry G(τ), and its half-width is equal to $3 \cdot 10^{-7}$ s. For this case, α = 0.96 and β = 0.87. However, at low temperatures T = 323 K and T = 293 K ($T < T_c$), this function becomes sharply asymmetric and cannot be described by the generally accepted HN function. The model needs to be complicated. The reasons for this complication, in our opinion, are the martensiticity of the semiconductor-metal phase transition and the large role of correlation effects in the process of completing the Mott part of the semiconductor-metal phase transition (Ilinskii et al. 2012).

Notably, such complicated processes are manifested in the presence of a series of maxima on the temperature dependence of the numerical value of the maximum of the function dN/dτ (Fig. 9). In this case, the maximum at T = 340 K is undoubtedly associated with the Peierls structural PPM, while the other maxima in Fig. 9 are due to the peculiarities of the Mott electronic semiconductor-metal phase transition at $T < T_c = 340$ K.

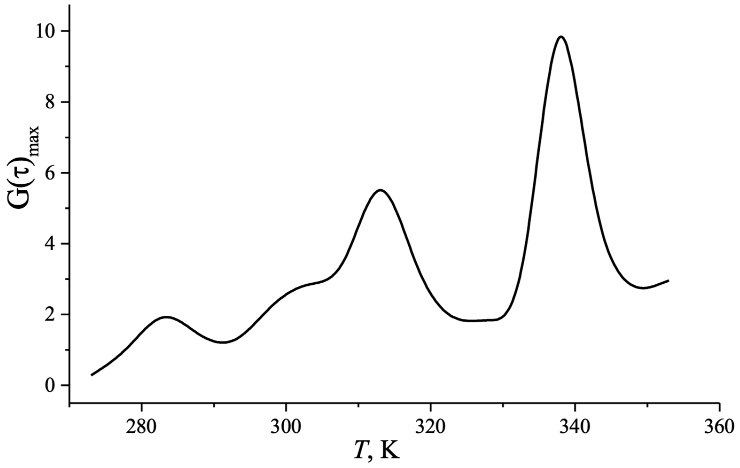

Fig. 9. Temperature dependence of the numerical value at the maximum of the relaxation time distribution function for the studied layers of vanadium dioxide

When describing the detailed reasons for the appearance of certain features in dielectric spectra (in particular, the asymmetry of the HN function) and the appearance of the reasons for such asymmetry, we proceed from the following physical considerations.

1) In the case studied by us, there is one type of relaxators which is realized in vanadium dioxide due to the relaxation process of screening of the external electric field by a gas of strongly correlated free electrons in the conduction band.
2) The parameters of the relaxation process of the dielectric response are significantly affected by the semiconductor-metal phase transition process, which is of a martensitic nature. Namely, the VO_2 film contains nanocrystallites of various sizes distributed according to a law close to a weakly asymmetric Gaussian (Ilinskiy et al. 2020). In this case, the width of the elementary hysteresis loops of individual nanocrystallites is inversely proportional to the square root of their diameter, and the phase equilibrium temperatures (the middle of the elementary loops) for all nanocrystallites are different, being shifted to different degrees towards low temperatures due to differences in the oxygen nonstoichiometry of nanocrystallites. Nonstoichiometry manifests itself in the formation of oxygen vacancies during the synthesis of films which have donor properties. They shift to the low-temperature side the point T_c of thermal equilibrium of the semiconductor and metal phases in a given nanocrystallite—that is, they shift the middle of its elementary loop. The nonstoichiometricity of large grains is much greater than the nonstoichiometry of small grains due to the effective healing of vacancy oxygen defects in small grains. Therefore, even at room temperature, a small part of the largest nanocrystallites turns out to be metallized—that is, at least the Mott part of

the VO_2 complex semiconductor-metal phase transition is completed in them into the metallic phase, which is due to the narrowness of their elementary loops.

3) The numerical value of the dielectric relaxation time is inversely proportional to the concentration of electrons in the conduction band. With an increase in the temperature of the semiconductor phase, an increase in the total concentration of free electrons occurs due to an increase in the number of metallized grains that have completed the Mott phase transition.
4) As is known (Ilinskii et al. 2020), the distribution of electrons in energy in such a strongly correlated material as VO_2 obeys the Migdal distribution, the mathematical expression for which contains the correlation energy, along with the Fermi energy, in the exponent. The correlation energy of electron-electron interaction consists of two parts: the energy of interaction of free thermally excited electrons of the conduction band with electrons of the atomic core, and the energy of direct Coulomb interaction of free electrons with each other.

At low temperatures of the semiconductor phase, the concentration of free electrons in individual nanocrystallites is low and strongly differs in nanocrystallites of different sizes, since the degree of their oxygen deficiency is sharply different, as indicated above.

This leads to a difference in the concentrations of free electrons in them and, accordingly, leads to a dispersion of relaxation times in nanocrystallites of different sizes. The long “tail” of the G(τ) function, which extends towards short relaxation times, thus reflects the “tail” of the size distribution of nanocrystallites in the film (Ilinskiy et al. 2020).

As the temperature of a set of nanocrystalites increases, these differences are smoothed out due to an increase in the degree of metallization—that is, an increase in the total concentration of free electrons, and an increase in the role of the direct Coulomb interaction between them (Apinyan, Kopeć 2015).

This manifests itself in a decrease in the numerical values of the relaxation times, and upon the transition to the metallic phase of most of the nanocrystallites of the film (for example, at $T = 353$ K), and in the complete symmetrization of the G(τ) maximum, since practically all nanocrystallites acquire the same concentration of free electrons and the same energy of direct Coulomb interaction between them. There only remains the fluctuation broadening of the maximum of the function G(τ) of the distribution of relaxation times over their numerical values.

Thus, an increase in the degree of total metallization of the film leads to a shift in the maximum of the G(τ) function of the spectral density distribution of relaxators over relaxation times towards shorter times, the G(τ) function becomes more symmetric, and its area and height increase, which is associated with an increase in the number of relaxators due to the involvement of new nanocrystallites of ever smaller sizes in the metallization process. The process ends at the temperature at which complete metallization of the VO_2 film occurs and the process of thermal growth of the concentration of free electrons saturates.

Conclusion

The work investigates the processes of dielectric relaxation in thin films of vanadium dioxide in the temperature range $T = 273–373$ K. The frequency dispersion of the dielectric permittivity and the presence of maxima in the spectra of dielectric losses are revealed, which indicates the existence of relaxation processes. The experimentally determined values of the relaxation parameters convincingly demonstrate the existence of a distribution of relaxators over relaxation times. According to the Cole-Cole model for the case of a symmetric distribution of relaxation times at high temperatures (at temperatures above the semiconductor-metal phase transition temperature), $\alpha = 0.96 \pm 0{,}03$ and $\beta = 1{,}00$. According to the Cole-Davidson model for the case of an asymmetric distribution of relaxation times (at $T < T_c$), $\alpha = 0.99 \pm 0{,}03$ and $\beta = 0.62 \pm 0{,}03$. The activation energy of relaxation processes turned out to be $E_a = (0.9 \pm 0.1)$ eV, which coincides with the numerical value of the band gap of vanadium dioxide at room temperature. The change in the nature of the dispersion of the dielectric permittivity, as well as the observed radical change in the numerical value of the G(τ) function at the maximum at $T = 340$ K, are due to the semiconductor-metal phase transition performed by VO_2 vanadium dioxide at a given temperature.

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