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FLUCTUATION NONDESTRUCTIVE TESTING OF SOLID MATERIALS

The paper presented analysis of experimental and theoretical studies of electric low-frequency fluctuations in solids. The possibility of extensive use of low-frequency fluctuations for solid material nondestructive testing is shown. Effectiveness of fluctuation nondestructive testing of solid materials is justified. The paper also highlights the applicability of fluctuation nondestructive testing to increase dependability of electronic devices.

Keywords: *fluctuations, noise, nondestructive testing, solid materials.*

The increasing complexity of modern electronic equipment and its functional importance, as well as the need to operate when subjected to various external factors requires high dependability of all components. This requires high quality materials to be used in electronic equipment. As electronic devices and equipment are manufactured mainly of solid materials, there is a need to develop methods of nondestructive testing of solid materials. For that purpose, the fluctuation non-destructive testing can be used. In this case, the quality of materials is evaluated by the behavior of fluctuation processes occurring in solids. Electrical low-frequency fluctuations are the most effective method to solve that problem. This is due to the following circumstances. Electric fluctuations can be quickly and easily measured. One type of noise, usually excess noise, dominates in different types of solids at low frequencies. Its spectral properties are well learned. Low-frequency excess noise is largely connected with structural defects in solids. The degree of imperfection can be evaluated according to spectral-response characteristics of the excess noise. The author examines the nature of electric fluctuations in solids and their application in nondestructive testing of electronic devices in [1-3]. The paper also analyzes the applicability of electrical low-frequency fluctuations in nondestructive testing of solid materials.

At low frequencies, electric fluctuations in solids are generally identified by excess noise that usually prevails over other types of noise in the low frequency range. Excess noise is a noise the power spectral density of which has the following form: $S(f)\sim 1/f^{a}$ where α is usually close to 1 (it is also referred to as noise $1/f$). Excess low-frequency noise has been extensively investigated and is well studied experimentally [1-5]. The frequency dependence and the order of magnitude of the noise spectral density are well known for many classes of solids and electronic devices. The properties of excess noise in a large number of different objects are experimentally studied. The origin of excess noise is associated with defects in the structure of solids. The findings of numerous studies indicate that as presented in [1-5]. Let us examine the experimental and theoretical studies that demonstrate the connection between excess noise and structure defects of solids.

Numerous and diverse experimental findings showing the connection between excess low-frequency noise and structure defects of solids have been obtained. Let us consider the most important of them. The earliest results in this area [6-8] showed that the plastic deformations of solids caused by pressure and high temperature increase the excess noise. Later, a large body of evidence of the impact of structural defects of solids on the properties of electrical low-frequency noise has been obtained. Let us consider the key findings of such studies per various types of solids.

In metals, one of the convincing arguments supporting the relationship between the mechanism of excess noise generation with structural defects is the dependence of the noise on mechanical deformations. In films of different metals such as lead, tin, platinum, gold, silver, an increase of the excess noise by approximately an order of magnitude with their deformation has been identified [9]. Upon relief of deformation, the noise level in the films decreased, but remained higher that the initial value. The observed effects may be associated with the formation and annihilation of structure microdefects. The results of the study of the spectral noise density dependence on mechanical stress in areas of elastic deformation for chromium film are given in [10]. With the increase of tensile stresses, reversible increase in the intensity of low-frequency noise is observed. This behavior can be explained by the fact that with growing tensile stress the distance between the atoms increases, thereby decreasing the activation energy for vacancy formation and increasing the concentration of defects of this type. The effects of plastic deformation on the chromium film lead to excess noise levels above initial values upon relief of the stress [10]. This can be explained by the appearance of additional microdefects as the result of plastic deformations.

It is known that the structural disequilibrium of vacuum condensates is largely due to own macrostresses. The dependence of excess noise on internal mechanical stress in thin aluminum films obtained by thermal evaporation in vacuum have been investigated [11, 12]. It was found that growing macrostress leads to the increase of noise spectral density. As it is known, tensile stress increases the concentration of vacancies, which explains the increase of excess noise level [12]. An increase of frequency exponent with growing positive and negative stress was also observed. The observed growth of the spectrum shape exponent with increasing mechanical stress indicates that in this case, the contribution of non-stationary mechanisms of noise initiation increases as well. Similar results were obtained for the films of chromium. Experimental results presented in [13] how that in chromium films there is a relationship between the level and nature of the excess noise and the quantity of internal mechanical stress. In the films with high internal stress a higher level of low-frequency noise can be observed. In addition, the spectrum shape exponent is higher for films with higher values of internal stress. Experimental study of relationship between excess noise and mechanical stress in the metal films showed that the dependence of noise spectral density of mechanical stress for films of aluminum, molybdenum and tantalum [12, 14, 15] obeys an exponential law. The growth of the noise spectral density with increasing stresses can be explained by intense growth of defect concentrations in films at sufficiently high mechanical stress. This is confirmed by the fact that electric resistance of the film reversibly increases when tensile deformations are applied.

The connection between the excess noise power with structural factors of metals was identified. The study of thin films of aluminum has shown [12] that the spectral density of the excess noise increases with the acceleration of film condensation, and this result was observed at deposition on substrates with substantially different physical properties. Moreover, it was found that the type of film production process (different aluminum deposition rate) affects their structure. The study of the microstructure of aluminum samples showed that a higher surface density of grains corresponds to a larger value of the noise spectral density. Similar behavior was identified in films of chromium, molybdenum, [10] and silver [16]. Thus, it was found that the dispersion of the crystallites leads to an increase of noise level. In chromium film, the intensity of excess noise was found to depend on the concentration of vacancies in the grains. These facts show the connection of excess noise with structural factors.

The effect of annealing on the excess noise was studied experimentally in films of aluminum [11, 17] and chromium [18]. Noise measurements were made before and after annealing of defects. It was shown that excess noise of films decreased as the result of annealing that lead to the reduction of structural defects. An investigation of the effect of annealing of induced defects on the low-frequency noise intensity in aluminum films is given in [19, 20]. Defects in the films were created by means of electron bombardment. After bombardment, a significant increase in noise intensity was observed. Annealing of defects led to the restoration of the low-frequency noise level. Paper [21] examines the influence of annealing on the excess noise in metal films and shows that the noise is caused by the motion of structural defects. Studies of excess noise in a large number of films of various metals [22] demonstrated that with increasing density of defects and impurities in the film the noise increases. It was established that the bombardment of copper films by electron beams leads to an increase of the excess noise spectral density by more than an order of magnitude. The study [23] has found that the radiation damage of indium-doped copper films cause an increase of the excess noise. The effect of γ-radiation on the excess noise in niobium films was studied in [24]. The impact of γ-radiation leads to additional defects in the crystal lattice. After the irradiation, the excess noise in the films increases. The reported results demonstrate the decisive role of metal structure defects in the generation of excess noise.

Numerous experimental facts support the connection between excess low-frequency noise and structural defects in semiconductors. Let us consider these results. The research of the influence of controllably introduced dislocations in silicon on the excess noise revealed the following. Paper [25] shows that the introduction of dislocations causes an increase in the noise level. The increase in noise strongly depends on the temperature. In [26] the introduction of dislocations led to a significant increase of the excess noise intensity, and the noise intensity increased monotonically with increasing dislocation density. The influence of defects on the noise were shown in experiments where structural imperfections were introduced by means of ion implantation. In [27,28] it was established that the structural damage caused by ion implantation leads to a significant increase in the excess noise in silicon. Subsequent annealing that restores structural perfection of the material causes a noise reduction.

It was established [29] that the ultrasonic treatment of epitaxial samples of GaAs (gallium arsenide) causes an increase of low-frequency noise intensity. A series of results has been obtained showing a connection between the excess low-frequency noise in semiconductors and structural defects arising under strong (destructive) compression. Semiconductor lattice disruptions arising in the destructive compression were well studied in [30, 31]. Various methods of studying the structure of solids show that the impact of mechanical stress on semiconductor materials leads to the generation of dislocations, declinations, vacancies, vacancy clusters and other types of defects. The study [32] presents the findings indicating the effect of compression on the destructive excess noise in GaAs samples. It was established that with the growth of destruction the intensity of low-frequency noise increases. The increase of the noise intensity was observed to reach two orders of magnitude. Due to the impact of mechanical stress a certain change in shape of the spectrum of the noise associated with the change of the frequency exponent is observed. It was found that under destructive compression the excess noise level increases to the same extent as the local concentration levels due to structural defects. The study of excess noise in silicon shown that with decreasing degree of structural perfection of the material the noise intensity grows [33]. These circumstances point out to an association of excess noise in semiconductors with structural defects.

An increase of low-frequency noise in semiconductors under the influence of radiation in the optical range has been detected. In [34] the effect of low-frequency noise spectrum readjustment in samples of GaAs under the impact of light was observed. It was also found that at low temperatures the effect of lighting results in a substantial increase in the noise level. The given explanation of this phenomenon is that the lighting significantly changes the concentration of minor carriers that being caught at permitted levels in the band gap can strongly influence the charge state of the trap centers and, consequently, the intensity and spectrum of low-frequency noise. Paper [35] studied the effect of laser radiation on the excess noise in crystals $Cd_xHg_{1-x}Te$. It was found that with the growth of irradiation energy the noise intensity increases. Accordingly, it is concluded that a higher noise level corresponds to an increased number of defects. That further confirms by the results of the study [36] that shows that defects cause excess noise in such materials. The discovered nature of the influence of optical radiation on the excess noise in semiconductors also points to a relationship between noise and structural defects.

An important evidence in favor of the fact that the origin of the excess noise is connected with the structural defects consists in the dependence of its properties on the surface condition. The surface of the material has especially many defects as it is the interface between volume and environment and, therefore, there is a high concentration of broken electron couplings due to the finiteness of sample geometric dimensions. Furthermore, some of these couplings can be filled by atoms of chemically active substances in the atmosphere which leads to chemical reactions on the surface. When studying the excess noise of germanium strings [37], it was found that upon replacement of ambient dry nitrogen with liquid nitrogen the noise increases significantly, and this is accompanied by a change in the frequency profile of the spectrum. Other studies established [38] that humid atmosphere may amplify excess noise by several orders of magnitude.

It was also found that the level of the excess noise was associated with adsorption and chemical adsorption of substances by the materials' surface. A similar phenomenon is observed in electron tubes and semiconductor structures. An overview such studies is given in [39].

Paper [40] established a correlation between excess noise of surface-barrier transitions with the dislocation density, which also confirms the influence of structural defects on the properties of the excess noise.

A number of experimental results showing the connection between low frequency noise in dielectrics with structural defects have been obtained. Electrical low-frequency noise in polymer dielectric films was investigated in [41-47]. A conclusive correlation between the level of excess noise and dielectric strength of polymer dielectric films was established in [41, 42]. It is known that the electric strength of dielectrics is related to the presence of defects. It was shown that the long-term exposure to alternating electric field causes a reversible noise increase in dielectric film, and therefore as a result of that increase, the correlation between noise level and dielectric strength becomes stronger [43]. The effect of damaging actions on the low-frequency noise in films was investigated in [44, 46, 47]. An irreversible increase of excess noise in dielectric films due to exposure to strong (destructive) electric fields, which indicates a relationship between noise and defects in films, was established [44, 46].

A variety of theoretical models, according to which the cause of the excess low-frequency noise are the defects in the structure of solids, has been developed. These models explain the origin of the excess noise in solids of various types. Let us examine the key premises of the explanation of excess noise from that perspective.

Let us consider the excess low-frequency noise in semiconductors. There are numerous theoretical models associates the origin of the excess low-frequency noise with structure defects of semiconductors. A large group of models associates the excess noise in semiconductors with the capture and emission of charge carriers by traps. In this case the excess noise is explained as follows. The defects can generate permitted levels in the gap band (traps). The capture of charge carriers by traps causes fluctuations in the number of free charge carriers which entails fluctuations of sample conductivity and electrical noise. Assuming that there is an even probability distribution of change of the charge state of the trap, then the spectrum of the fluctuations caused by one trap has the form of the Lorentzian spectrum. In a semiconductor, there is a set of traps with different time constants . It is believed that the traps are independent. The spectrum of fluctuations in the number of free carriers in a semiconductor is described by the following expression

$$S(f) \sim \int_{\tau_1}^{\tau_2} \frac{\tau g(\tau)}{1+4\pi^2 f^2 \tau^2} d\tau, \tag{1}$$

where $g(\tau)$ is the density distribution of time constants. Provided that $g(\tau) \sim 1/\tau$ in the frequency band $\frac{1}{2\pi f \tau_2} << f << \frac{1}{2\pi f \tau_1}$ fluctuation spectrum obeys the law: . Thus is $S(f) \sim 1/f$ explained the excess noise due to the processes of capture and emission of charge carriers on traps. Noise generation with the spectrum of the type $1/f$ is based on the superposition of processes on traps having a broad distribution of time constants. Specific physical mechanisms that substantiate that explanation of excess noise may be associated with the activation and tunneling of carriers into bound states on traps. An extensive overview of these theoretical models is presented in [2-4]. As of today, the theory connecting the explanation of excess noise with carrier capture by traps is the most important in semiconductor studies.

Electrical fluctuations in semiconductors caused by capture and emission of charge carriers by structural defects are more generally analyzed by the author in [2, 3, 48, 49]. The fluctuation process has been analyzed, when the probability of a carrier capture by the trap is statistically associated with the trap empty time, and the probability of the carrier emission is statistically associated with the bound time on the trap; the statistical relationships are defined in general terms. As a result, the following expression for the spectrum of low-frequency noise in the semiconductor caused by traps was obtained:

$$\frac{S(f)}{I^2} = \frac{\nu}{NV} \sum_{i=1}^{l} \frac{\sigma_i N_i}{1+\frac{1}{g_i} e^{-\left(\frac{E_i - E_F}{kT}\right)}} \Phi_i(f), \tag{2}$$

here I is the current in the sample, v is the average thermal velocity of carriers, N is the number of carriers in the sample, V is the sample volume, σ_i is the trap capture cross-section, N_i is the number of traps of each type, l is the number of trap types in the sample, g_i is the degeneracy factor, E_i is the trap energy, E_F is the Fermi level, k is the Boltzmann constant, T is the temperature, $\Phi_i(f)$ is the function defining the dependence of the spectral density of fluctuations on the frequency associated with distributions of the trap empty and filled times. This mechanism allows generating noise with a spectrum $1/f$.

Paper [50] suggests a model of excess noise in semiconductors linking it with the fluctuations of the energy levels' population in the tail of density-of-states function penetrating the gap band of the semiconductor. The cause of the electrical noise in this case are the fluctuations in the concentration of free carriers in heavily doped semiconductors caused by exchange of carriers between the conduction band and the levels of the tail. The formation of tails of density of states falling into the depths of the gap band is a result of semiconductor structure imperfections. These are impurities, defects, local stress of lattice and so on. In this model it is assumed that the level of the capture cross-section of the tail level σ_n decreases exponentially with increasing energy ε. Time constant $\tau_0(\varepsilon)$ of capture per level with energy ε:

$\tau_0(\varepsilon) \sim \frac{1}{\sigma_n(\varepsilon)}$, consequently $\tau_0(\varepsilon) = \tau_{00} e^{\varepsilon/\varepsilon_1}$ where τ_{00} is the capture time constant at $\varepsilon=0$, ε_1 is the constant defining the capture cross-sections decrease with increasing energy ε. It was assumed that the tail of the density of states decreases exponentially into the depth of the gap band as $\rho = \rho(0)e^{-\varepsilon/\varepsilon_0}$, where ρ is the density of states, ε_0 is the constant characterizing the droop rate of the density of states. In the case where with the increasing energy the time constant τ_0 is growing faster than the density of states decreases, and the temperature is relatively high, the low-frequency spectral density of the relative fluctuations of the free carrier concentration (and, correspondingly, the relative spectral density of fluctuations of the sample resistance) has the following form:

$$\frac{S_R(f)}{R^2} = \frac{S_n(f)}{n_0^2} \approx \frac{4N_0 e^{-\varepsilon_F/\varepsilon_0}}{VN_d^2(\tau_{00}e^{\varepsilon_F/\varepsilon_1})^{\Gamma-1}} \frac{kT}{\varepsilon_0} \frac{1}{(2\pi f)^{\Gamma}}, \tag{3}$$

where $N_0 = \int_0^{\infty} \rho(\varepsilon)d\varepsilon, \Gamma = 1 - \varepsilon_1/\varepsilon_0 - \varepsilon_1/kT$, ε_F is the Fermi level, n_0 is the equilibrium concentration of electrons, N_d is the concentration of donors, V is the sample volume. Provided that $\varepsilon_1 << \varepsilon_0$ and $\varepsilon_1 << kT$ noise spectral density varies as 1/*f*.

Excess noise due to fluctuations of the charge carrier concentration can be observed in systems with hopping conduction mechanism. The spectral density of the resistance fluctuations of lightly doped compensated semiconductor in the temperature range at which the conductivity is of hopping nature was calculated in [51]. Frequency of carriers' tunnel hopping between two doped centers is exponentially dependent on the distance between them: $\nu(r) = \nu_0 e^{-2r/a}$, where α is the effective Bohr radius, v_0 is the factor. Because the distance between the defect centers is a random variable, the semiconductor under the hopping conduction represents a disordered medium. At frequencies that are low compared to the hopping frequency of charge carriers on critical grid (infinite cluster determining the conductivity of the semiconductor) $f << \nu$, conductivity fluctuations are associated with fluctuations in the number of carriers on the critical grid. The spectral noise density rises with decreasing frequency and is related to the concentration of impurities. In a wide frequency range spectral density of conduction fluctuations is expressed as $S(f) \sim f^{-\alpha}$, where the exponent α<1 and depends on the value Na^3 (N is the concentration of impurities). In the limit of very low concentrations ($Na^3 \to 0$) the noise spectral density increases with decreasing frequency according to the law, approaching 1/*f*. However, with the realistic values Na^3 of the exponent α is significantly less than 1.

There are numerous approaches that associate the origin of excess low frequency noise in metals with structural defects. Among various types of defects the most essential to metals are vacancies, since their formation and migration requires relatively little energy. In this regard, models linking noise with vacancies is of particular interest to explain the excess noise in metals. Models exist which the low-frequency noise in metal films is conditioned by resistance fluctuations due to fluctuations in the number of vacancies in the sample. The lifetime of vacancies is a random variable and is determined by the average distance between sources (drain nodes) of vacancies. In the model developed for homogeneous metals [52] drain nodes are uniformly distributed throughout the volume. In this case, the probability density for each vacancy destruction during its lifetime is constant. Events of generation and annihilation of vacancies are statistically independent, and the average vacancy settled lifetime is given in the following expression: $\tau_{\nu 0} = \tau_0 \exp\left(\frac{E_\nu}{kT}\right)$, where E_v is the activation energy of a vacancy generation. The power spectrum of the noise generated when a current I_0 flows through a homogeneous metal sample with the average number of vacancies N_v has the following form [52]:

$$S_u(f) = 4\overline{\delta R^2}\, I_0{}^2 N_v \frac{\tau_{v0}}{1+4\pi^2 f^2 \tau_{v0}^2}, \tag{4}$$

where R is the resistance of the sample. In real metal films due to the heterogeneous distribution of drain nodes in the sample there is a wide range of relaxation times associated with the mechanism of generation and annihilation of vacancies, which explains the type of noise $1/f$ in a wide frequency range. Thus, as follows from formula (4), the noise increases with the number of vacancies.

Another sufficiently common approach to the explanation of the excess low-frequency noise in metals is the concept that associates the noise of type $1/f$ with internal friction. At low frequencies, the movements of defects create the friction reorientation, migration, etc. Accordingly, the low-frequency noise caused by internal friction can be revealed in a variety of metals and be associated with defects in their structure. The possibility of electrical noise spectrum of type $1/f$ due to internal friction is considered in [53]. Fluctuations caused by the random nature of reorientation of defects have been analyzed. The reorientation of defects whose symmetry is lower than the point symmetry of the crystal causes a change in the scattering of electrons on them. Consequently, fluctuations of the electric resistance of metals take place. If there is a distribution of the concentration of defects $n(E)$ in activation energies E such that the relaxation time of defects is expressed as $\tau = \tau_0 \exp\left(E/kT\right)$, then the electrical noise with a spectrum close to $1/f$ occurs, namely:

$$\begin{aligned} &\frac{S_U(f)}{U^2} \approx \frac{n(E_\omega)[l\sigma_s(E_\omega)]^2 kT}{Vf}, \\ &E_\omega = kT\ln(\omega\tau_0)^{-1} \end{aligned} \tag{5}$$

where σ_s is the electron scattering cross-section by defect, l is the electron mean free path, V is the sample volume. Thus, the given mechanism of conductivity fluctuation can explain the noise with a spectrum of type $1/f$ in metal films.

Summarizing the experimental and theoretical studies, we can draw the following conclusions. The results of numerous studies show that excess low-frequency noise is associated with structural defects in solids. Those results were obtained in the study of different types of solids. Experimental studies have repeatedly demonstrated the connection between the spectral characteristics of the excess noise with the structural defects. The most important facts are as follows. The excess noise grows with increasing density of impurities and structural defects. Noise amplification takes place due to mechanical deformations both in the areas of plastic and elastic deformations. There is an increase of noise due to the impact of penetrating radiation. Growth of the intensity fluctuations is observed due to excessive exposure to strong (destructive) electric fields. There is an increase of noise level caused by optical emission. Ultrasonic treatment affects the intensity of fluctuations. Noise reduction occurs as the result of annealing that decreases structural defects. Excess noise strongly depends on the samples manufacturing technology.

Theoretical models for different types of solids, whereby the origin of the excessive low-frequency noise is conditioned by structural defects have been developed. These models allow explaining many results of experimental studies of this type of noise. The experimental and theoretical studies of excess low-frequency noise show its relation to structural defects and allow determining the dependence of noise spectral characteristics on the number of defects. In addition, excess noise may be associated with the speed at which the number of defects grows [54]. Thus, the spectrum of excess noise contains information on the degree of solid material defectiveness and may contribute to the evaluation of the rate of degradation of its structure. Consequently, the excess low-frequency noise can be effectively used to assess the quality of solid materials.

Thus, electrical low-frequency fluctuations can be used for nondestructive testing of solid materials that are the foundation of electronic products. Therefore, the fluctuation nondestructive testing can be used to improve the dependability of electronic devices. This nondestructive control method has the following advantages. In many cases, the electrical low-frequency fluctuations provide more complete information regarding the defects of solid materials structure than it is possible with other methods. Usually, excess noise dominates over other types of noise in the low-frequency range. This allows reliably evaluating the noise characteristics. High sensitivity spectroscopy of fluctuations enables high-precision noise spectra measurement. Noise measurement can usually be carried out sufficiently fast. The above facts highlight the high efficiency of the fluctuation nondestructive testing of solid materials.

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