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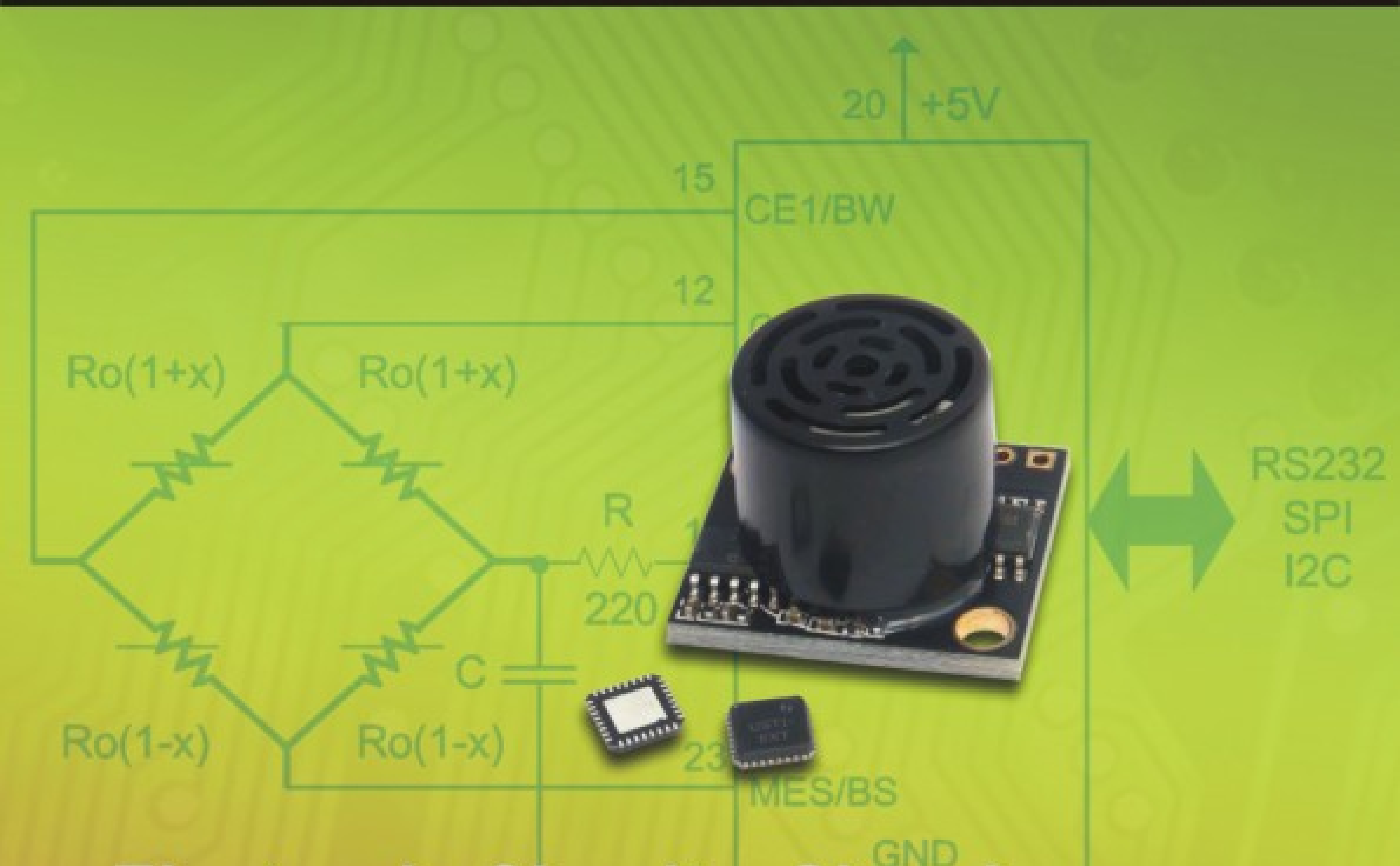

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Contents

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Editorial

IFSA Publishing Starts to Publish Hardcover and Paperback Books
Sergey Y. Yurish, Editor-in-Chief I

Research Articles

Research in Nanothermometry Part 4. Amorphous Alloys of Thermo-resistive Thermometry
Bohdan Stadnyk, Svyatoslav Yatsyshyn, Pylyp Skoropad.......... 1

Research in Nanothermometry. Part 5. Noise Thermometry and Nature of Substance
Svyatoslav Yatsyshyn, Bohdan Stadnyk, Zinoviy Kolodiy.......... 8

Design of Linearized Thermistor Connection Circuit Using Modified 555 Timer
Narayana K. V. L. and Bhujanga Rao A........... 17

Design and Development of Microcontroller Based Photoacoustic Spectrometer
P. Bhaskar, Immanuel J., and Bhagyajyoti.......... 26

The Design of a New Instrument for Pen-contact Force Information Acquisition During Handwriting
Jianfei Luo, Baoyuan Wu, Qiushi Lin, Zhongcheng Wu, Fei Shen 35

ARM Cortex Processor Based Closed Loop Servo Motor Position Control System
Madhusudhana Reddy Narayanareddygari, Nagabhushan Raju. K, Chandra Mouli. C., Chandrasekhar Reddy Devanna 45

The Hardware Design Technique for Ultrasonic Process Tomography System
Mohd Hafiz Fazalul Rahiman, Ruzairi Abdul Rahim, Herlina Abdul Rahim and Nor Muzakkir Nor Ayob 52

Design, Development and Testing of a Semi Cylindrical Capacitive Sensor for Liquid Flow Rate Measurement in Process Industry
Sagarika Pal, Sharmi Ganguly 62

Synchronization Based SAW Sensor Using Delay Line Coupled Dual Oscillator Phase Dynamics
Shashank S. Jha and R. D. S. Yadava 71

Intelligent Robust Nonlinear Controller for MEMS Angular Rate Sensor
Mohammad-Reza Moghanni-Bavil-Olyaei, Ahmad Ghanbari, Jafar Keighobadi. 92

Analysis of the Self-Calibration Process in a Displacement Sensor in Applications of Hip or Knee Implants
Shiying Hao 106

Acoustic Detector for Determining the Type and Concentration of a Solution
Tariq Younes 119

Low Concentration Sodium Chloride Salinity Detection System
Hee C. Lim, Hio Giap Ooi, Yew Fong Hor. 127

ARM Processor Based Embedded System for Examination Question Paper Leakage Protection System
Jyothi Pattipati, Chandra Mouli Chakala, Chaitanya Pavan Kanchisamudram, Nagaraja Chiyedu and Nagabhushan Raju Konduru 134

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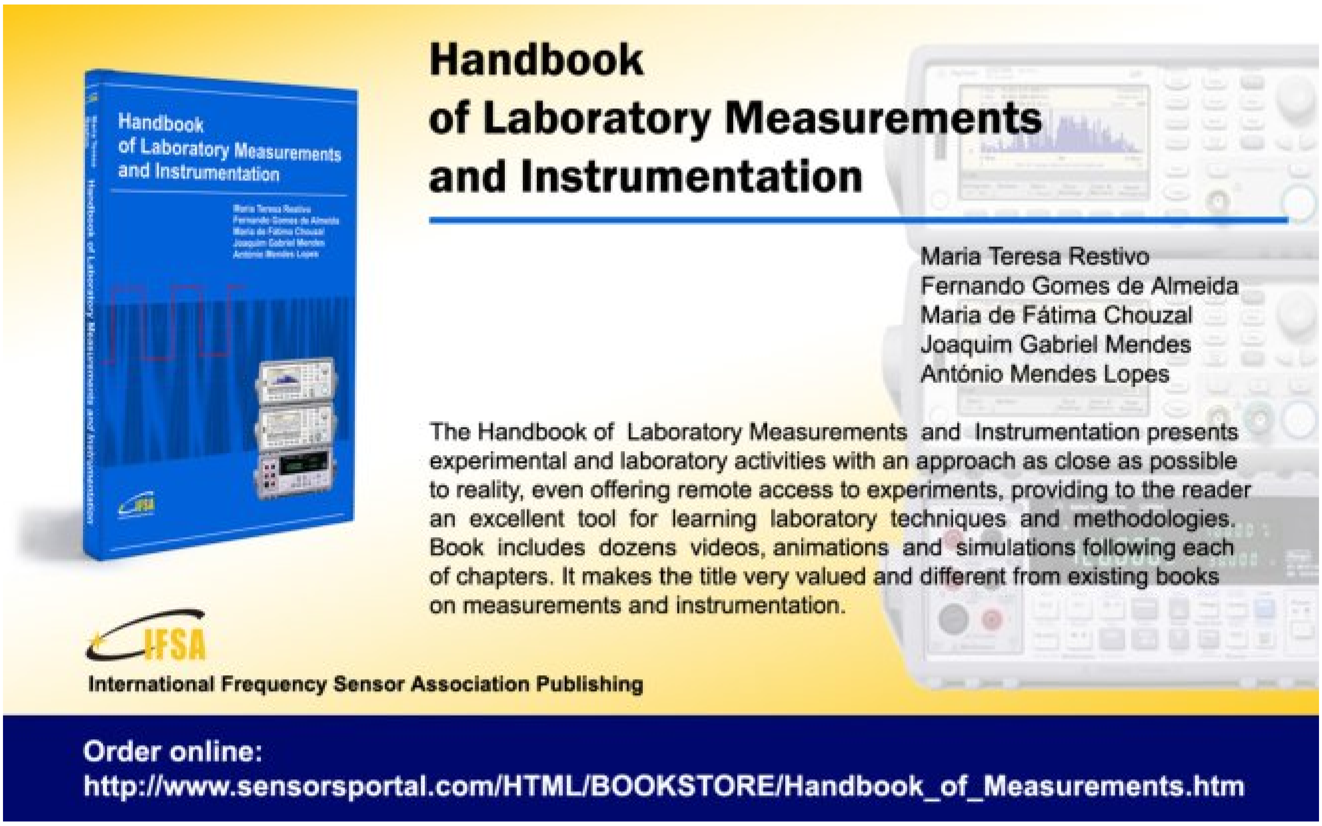

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Research in Nanothermometry.
Part 5. Noise Thermometry and Nature of Substance

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Abstract: More precise noise thermometer with different sensitive elements demonstrates the opportunity to characterize the features of matter at the micro- and nanoscale. The calibration characteristics in a wide frequency and temperature range are investigated. *Copyright © 2012 IFSA.*

Keywords: Temperature measurement, Calibration characteristics, Electrical noise, Frequency band, Structural defects of thermosensitive substance.

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1. Introduction

Nanotechnology has not arisen at the empty place but is the natural upshot of the scientific cognition directed into the depth of substance. Micro-, nano- dimensioned and structured materials evince new totalities of properties and functions in comparison with the bulk analogs. Their retrievable research and production are assured by the development of nanometrology, and first of all, the methods and means of temperature measurement, since any physical property is meaningful at some controlled temperature. That is why all sorts of measurement are based on the temperature monitoring, and in general temperature measurements make ~50 % of a total amount of measurements. The number of direct methods includes 5 methods of thermometry: *gas, acoustic, optical, magnetic and noise* [1]. Those methods are based on the fundamental physical laws whose mathematical descriptions comprise the thermodynamic temperature.

A noise method is increasingly applied in Nanotechnology [2] and Nanobiotechnology [3]. It has been implemented in the investigation of the electrical noise [4], particularly $1/f^{\gamma}$ noise [5]. Let us distinguish the monograph [6], where the estimation of the structure discreteness was connected with electrical noises as manifestation of the energetic fluctuation dissipation in substance. Noise thermometry is the metrorological method built on the use of relation of noise electrical power and temperature. Therefore it is considerably more precise and consequently informative.

On the one hand, the absence of the holistic approach to the description of phenomena and, on the other hand, the lack of metrological elaboration of a noise method do not promote its reasonable use in Nanotechnology. Therefore, research results are not interpreted comprehensively that is not conducive to further progress in Nanotechnology.

2. Aim of the Work

Goals of the work consist in the detailed research of the calibration characteristics of the noise thermometers (NT) with sensitive elements made from different thermometric substance (metals, alloys, composites) calling for scientific and design investigations aimed at developing noise thermometry for experimental verification of nanotechnology processing and applying modes including the influence of substance structural defects.

3. Experimental Investigations

Objects of investigation include 100 Ohm (at 27.15 K) sensitive elements of NT made from pure metals - *Ni, Pt, Cu*; alloys — nichrome and composites of various oxides. The research has been performed in the reference temperature points ITPS - 4.2 K, 77 K, ... 273.15 K - according to the IMECO method, and at higher temperatures. The obtained results have been processed using the Curvescript software.

3.1. Deviations from the Calibration Characteristics of the Thermometer at 77 K

In [7], an average, relative power of spectral density (PSD) P_{el} linearly depends on the temperature T in the area of the studied temperature. This means that experimental characteristic of NT is as follows:

$$T = bP_{el}, \tag{1}$$

where b is the coefficient. The error of the determined temperature (instrumental error of NT) is:

$$\delta T = \delta b + \delta Pel \tag{2}$$

The NT calibrating results at 77 K are shown in Fig. 1 as $\delta T = \delta T\ (K),$ where K is the characteristic parameter, determined below. The revealed deviations from linearity of calibration characteristics as the relative error δT have been described by (2). Deviations increase from copper (0.05 %) to nickel (0.26 %) sensitive elements. The mentioned deviations are not fixed for elements made from the transition metal alloys and composites.

3.2. Investigation of Calibration Characteristics under Stress-influence

The research is conducted on the *Ni-Cr* sensitive element. The letter is wound on a conical (20 : 1) matrix and consequently deformed. The linear elongation is carried out at 77 K. At the same time the electrical resistance and its changes δR, and relative PSD of electrical noise and its changes δPel

(Fig. 2) are measured. Results testify that: a) power changes and, therefore temperature changes at 77 K are proportional to the relative elongation ε of a sensitive element; b) larger values of deformation result in nonlinearity influences of the calibration characteristic δPel. That may show effect due to the changes in the electrical resistance δR, more intensively as compared to the changes in electrical power.

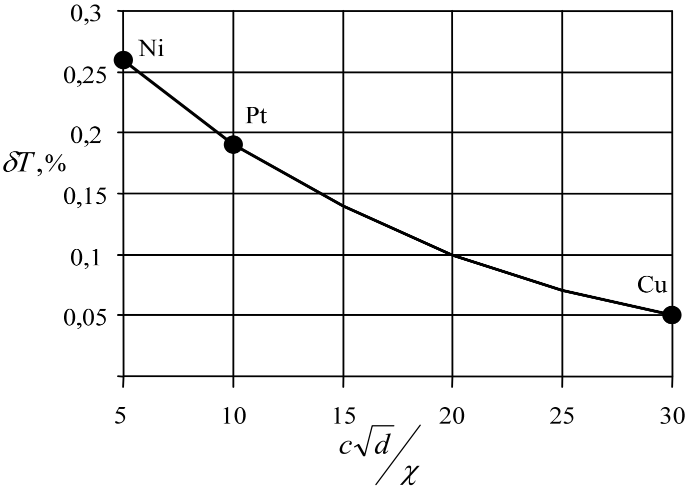

Fig. 1. The instrumental errors $\delta T = \delta T\left(c\sqrt{d}/\chi\right)$ of noise thermometers with sensitive elements of *Cu*, *Ni, Pt* at 77 K: theoretical dependence (—) and experimentally defined values (·).

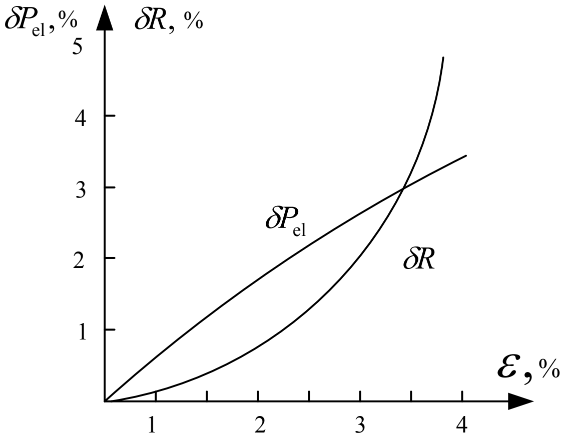

Fig. 2. Changes in calibration function δP_{el} and electrical resistance δR of *Ni-Cr* NT due to its elongation ε.

3.3. Frequency Dependence of Electrical Noise Power

The known Nyquist formula is equitable for thermal electrical noise at frequencies higher than 1000 Hz and for sensitive elements, made from alloys and composites. Calibration characteristics of NT with sensitive elements, made from alloys, are linear with no recorded deviations from the linearity. At lower frequencies, so-called $1/f^{\gamma}$ noises whose nature has not been studied fully are fixed.

4. Theoretical Investigation

In process of temperature measurement with the help of thermotransducers, the neglecting by the changes in the thermodynamic state of thermometric substance seems to be ordinary but not fully correct practice. Depending on the type of an inner substance state and external environments, the thermodynamic computation serves as a base for evaluating the characteristics and their changes. The substantial temperature dependence of mechanical properties has complicated the situation. It is shown up not only in an eventual mechanical refusal, but also in the drift of calibration characteristics. The latter one acquires the main significance of thermometry.

The consideration is important for such an ordinary factor or influence on metrological characteristics as a statistical-deformation factor that increases at the combined action of temperature and strains (structure, elastic, plastic or others) in thermometric substance during measuring. This factor has conditioned in the local distortions of temperature in the mentioned materials as a result of tensile micro concentrator actions, which are considered as quasi local nanosized defects of thermo fluctuation origin. The given distortions substantially have lowered the real durability of thermometric substance as to its theoretical values through electromechanical noises, which at the same time stipulates the deviations of NT calibration characteristics and worsening in its precision.

4.1. Processes in Thermometric Substance, and the Instrumental Error of a Noise Thermometer

Analysis of a measurement error has shown the additional component existence (Fig. 1) that goes beyond a basic acceptable error. This error is caused by structural processes in thermometric substance [8]. The constancy of temperature - 77 K – does not mean the thermodynamic equilibrium state of a sensitive element and environment. The state changing is an aging result. The accumulation of excess energy caused by the sensitive element manufacturing (bending, tension) causes inner strain and a consequent nonequilibrium thermodynamic state in thermometric substance. Their relaxation depends on several factors (temperature, time, type and concentration of defects).

Otherwise, the condition of thermodynamic equilibrium, at which formula of Nyquist is derived has been broken in the case of a real NT. The temperature dependence of electrical noise power is derived directly from the basic equation of thermodynamics. In the stationary nonequilibrium state, the thermometric substance calibration characteristic nonlinearity appears due to violation of energetic (entropy) processes of environment-substance exchange. It is expressed by the instrumental (absolute and relative δT) error as:

$$\Delta T = T_c - T_r = \left(b_c - b_r\right) P_{el}, \;\; \delta T = \frac{b_c - b_r}{b_r} = \frac{\Delta b}{b_r}, \tag{3}$$

where T_c and T_γ are the estimated and real temperature, respectively; b_c and b_γ are the constants of estimated and actual calibration characteristics. The prolonged use of a thermometer leads to maximizing the constant $b \sim (dS / dt)^{-1}$ at minimizing the entropy dissipative flow [6]:

$$\frac{dS_\vartheta}{dt} = -\frac{\Delta S}{\tau_p} = \min .$$

The nonstationary nonequilibrium thermodynamic state is connected to the second component of the equation (2), and corresponds to the power change of nonequilibrium electrical noise in the thermometric substance.

Let us consider the details of the origin and manifestation of influence of a thermometric substance state on electrical noise. A sensor is wound at 300 K and used at low temperatures. Therefore its relaxation effects of elastic-plastic deformation lead to the emergence of the error.

The substance of the density ρ rapidly releases the previously accumulated elastic energy with appearing the microcracks of the length *2l*. Thus, the relaxation constant τ_1 is estimated as $\tau_1 \sim l\sqrt{{2\rho E}/{\sigma^2}}$, where ${\sigma^2}/{2E}$ is the density of elastic energy. The latter can be transformed into surface microcracks energy with its relaxation constant τ_2: $\tau_2 \sim l\sqrt{{\rho l}/{\gamma}}$, where γ is the specific energy of surface tension; or removed from the relaxation place with the constant τ_3, linked to the thermal diffusivity *a:* $\tau_3 \sim {l^2}/{a}$.

It should be noticed that at the temperatures lower than 20-30 K, thermal diffusivity in 100 and more times is higher than at 300 K. So τ_3 is much smaller in comparison with the relaxation constant of motion τ_4 ($\tau_4 \sim {\lambda}/{\omega}$) or reproduction τ_5 of dislocations ($\tau_5 \sim \left(\frac{\rho l^3}{nL_d E_d}\right)^{1/2}$), where λ is the effective length of a dislocation run; ω is dislocation velocity; L_d is the typical size; E_d is the dislocation energy, referred to one interatomic distance.

The effect of the mentioned above constants $\tau_1 \ldots \tau_5$ is combined, and depends on the temperature and background of material, forming the total relaxation constant $\tau_{n.st} = \frac{1}{\sum_{i=1}^{n} \frac{1}{\tau_i}}$. Consideration of competitive effects of the constant τ_2 due to microcrack formation, and constant τ_3 due to a heat removal from the place of energy relaxation produces the modified constant $\tau_{st} = \frac{\tau_2 \tau_3}{\tau_2 + \tau_3}$, .

The joint effect of these two mechanisms creates the reasons for changes in the electrical noise power and thus changes in the readings of NT. Hence, the methodical error of NT, whose sensitive element is in a stationary nonequilibrium state, is determined by the competitive action of two major in those conditions dissipation processes that form deviation from the calibration characteristics of [9] (Fig. 1):

$$\delta T = \tau_2 / \tau_1 = A(ad)^{1/2} c/\chi , \tag{4}$$

where C is the sound velocity; m is the grain size; d is the atomic size, χ is the thermal diffusivity. (In [10] τ_2/τ_1 is criterion of substance plasticity: the higher is this value, the greater is material plasticity). Hence, the lower speed of sound and higher thermal diffuseness, the more efficient work mechanism for a heat removal and the less noticeable influence of dislocations on the electrical noise power and consequently on a NT error. The described before concerns pure metals and is not related to alloys and composites due to significance of the process of dislocation multiplication (constant τ_5) that occurs in their blades and is accompanied with the microcrack formation.

The average and high temperatures are characterized by the relaxation mechanism that is based on the motion and multiplication of dislocations with the constant τ_5. Finally, high temperatures up to a melting point are matched with diffusion removal at the relaxation constant τ_4 at is, in the high-temperature case, one should consider the competitive action of two relaxation mechanisms: diffusion mechanism and formation of microcracks in the deformed local substance microvolumes. The role of diffusion is emphasized by the research of $1/f^{\gamma}$ noise.

The higher self-diffusion coefficient, the longer must be the microcracks formed due to increased substance plasticity with this value of mechanical stresses. In so doing, the smaller cracks as compared to critical, are quickly healed due to diffusion of vacancies from the area of microcrack to nearby located sinks. The introduced plasticity criterion is varied at the high temperatures to:

$$\tau_4/\tau_1 \sim (ad)^{1/2}\, c/D\,, \tag{5}$$

since in (4) the thermal diffusivity χ is replaced by the coefficient of diffusion D which increases exponentially with temperature. It means practical absence of possible deviations of calibration characteristics at high temperatures.

4.2. Nature of Electrical Noise

It is known that the observed electrical and other stochastic fluctuation systems are characterized by PSD $S_{\omega}(f)$, proportional to $1/f^{\gamma}$. This is a flicker-noise or a noise type $1/f^{\gamma}$. Experimental data have revealed that their PSD could be defined as:

$$S_{\omega}(f) = \frac{\alpha}{f^{\gamma}}, \tag{6}$$

where α is the constant; γ=0...3. For instance, our research has concluded γ=2.8 at the frequency band 3-12 Hz; γ=0.5 at 12-17 Hz for *Pt*; γ=2.28 at 3-12 Hz and γ=0.9 at 12-17 Hz for an oxide resistor. Considering the problem of thermal and low-frequency noise types $1/f^{\gamma}$, we would discuss the peculiarities of electron-phonon interaction by applying different approximations, regarding the possible types of adequate descriptions.

The case of equilibrium noise (current does not pass through the sensors). The thermal noise power dissipation caused by the appearance of microcracks at the frequencies higher than 100 Hz. Here, the energy is accumulated in quasi defects and transformed into microcracks, strengthening the intensity of noise. These defects disintegrate with sound, light and electron emission, becoming a point source of dislocations, vacancies and so on. In thermometric substance, the field of interrelated thermo fluctuational local tensile quasi defects causes the uncorrelated nature of thermal noise. Energy concentration and dissipation are realized in these quasi defects of vacancy origin through their interaction with phonons. On passing the alternating current, a large number of phonons, accumulated on the mentioned defects, are relaxing in a reversible or irreversible manner, causing, accordingly, $1/f$ or thermal electrical noise. The reversibility means disappearance of these defects and appearance of phonons.

With the help of frequency dependencies of PSD, the analysis [11] reveals the significant effect of dissipating the energy, accumulated before. The frequency dependent noise within the low-frequency range (below 80 Hz) corresponds to the reverse transformation of phonon energy in tensile defects. Their PSD will be $S(f) = P_{el} / \Delta f = c/af$. At this frequency band, the action of phonons on defects leads to scattering their energy, for example, by vacancy movement without the appearance of microcracks. The transition from $1/f$ noise to thermal noise occurs at the critical frequency, dependent on temperature. At low temperatures, a dominant heat dissipation effect should be considered. Therefore, the critical frequency changes from $\Delta f/f = d/\Lambda$ at medium temperatures to $\Delta f/f = (a/d)^{1/2}\,\chi/\Lambda c$ at low temperatures (where Λ is the length of a micro concentrator). For high temperatures when the role of diffusion is essential, the expression changes to $\Delta f/f = (a/d)^{1/2}\,D/\Lambda c$.

The previously conducted experimental studies of calibration characteristic changes emphasize the role of mechanical stresses in the investigated substance but do not interpret the mechanism of their action. That is why the results of specific studies are involved to evaluate the influence of mechanical micro concentrators and local distortions of temperature on the properties of thermosensitive substance. Power fluctuations are supposed to be the factor promoting the transformation of the energy applied to the thermometric substance into microcracks. Local temperatures of tensile defects are the same (~ 950 K) at the moment of rupture which forms a significant argument for the benefit of defining role of tensile micro concentrators (Fig. 3). The mechanical stress is not acting directly, but via the deformation, causing the heating of the mentioned quasi defects.

The case of non-equilibrium noise. There are other cases of non-equilibrium noise in films whose structure is different from thermodynamic equilibrium: freshly made films, deformed or irradiated films. It is known in the technical literature (4) that for *Cr* films on the glass substrate, the frequency dependence of relative PSD is a function of mechanical stresses in an elastic deformation area (relative film deformation by 0.4 % and more corresponds to a plastic deformation area where irreversible changes of noise are observed). As the average values of tensile stresses in the film are increasing from 100 MPa (without external force application) to 300 MPa, PSD of $1/f^{\gamma}$ noise increases about 3 orders, and the spectrum shape factor γ increases from 1 to 2.5 with removing external bend force. PSD of $1/f^{\gamma}$ noise for the area of elastic deformation returns to its initial state.

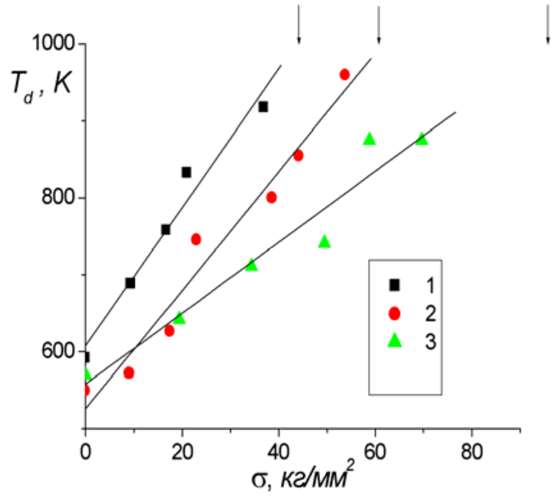

Fig. 3. Local temperature of tensile defects vs. external mechanical stresses at 300 K for plastics of different long-term durability σ: 1 – 40 kg/mm²; 2 – 60 kg/mm²; 3 - 110 kg/mm² (arrows — breaking point) [12]

4.3. Research Peculiarities, and Imperfection of Result Processing

When comparing the results of research - experimental and theoretical - it becomes evident that the character of frequency dependence at 12 Hz and less for the platinum and oxide resistor differs significantly by the value of spectrum shape factor γ. There could be several reasons.

A. The first one consists in: the measuring and processing of experimental results suggest the invariance of PSD noise $S'_\omega(f)$, cut by a filter within the certain frequency band Δf. Thus, it does not take into account that PSD $S_\omega(f)$ is represented by the expression:

$$S_\omega(f) = \lim_{\Delta f \to 0} \frac{P_{el}(f, \Delta f)}{\Delta f}, \tag{7}$$

where $P_{el}(f, \Delta f)$ is the PSD at the frequency band from $f - \Delta f/2$ to $f + \Delta f/2$ that has reduced to the really used approximate expression:

$$S'_\omega(f) \approx \frac{P_{el}(f, \Delta f)}{\Delta f} \tag{8}$$

As a result, there appears the additional error caused by the linearization of the expression (7) by (8). It significantly strengthens the character of the frequency dependence of PSD at approaching frequency to zero. The conducted analysis for the spectral distribution of PSD by Debye model has shown that this error $\delta S_\omega(f) = C/f^2$ is methodical. That is, the measured dependence of PSD noise *1/f* is quadratically related to the frequency. Einstein model where the temperature dependence of PSD is absent (the case of thermal noise) allows to get rid of the methodical error $\delta S_\omega(f) = 0$.

B. The second case and, accordingly, the other component of the NT instrumental error arise due to the improper technique or means of measurement. Thus, without the possibility of increasing the integration time or bandwidth of expansion for a selectivity filter, it results in the dependence, close to the cubic frequency PSD $S_\omega(f)$ -dependence. It is caused by Hrenander uncertainty principle:

$$t\Delta f = Const, \tag{9}$$

According to it, narrowing the bandwidth Δf of a filter requires a longer duration t of measuring noise, thus there remains the same referred component of an error. The shortening of the bandwidth of a filter at the fixed duration results in the significant uncertainty of the measured PSD.

5. Conclusions

1) It was shown for noise thermometers with sensitive elements, manufactured from different materials that the intensity of electrical noise substantially depends on different transport processes in the thermometric substance. For instance, at 77 K the most significant are the transfer processes of heat and charge. At the certain temperatures and deformations, the importance of other processes that may become crucial is growing. That is the case, for example, of selective gas sensing with the pristine graphene [13], where some gases induce clear peaks in the well reproducible noise spectra, which could be used as their signatures in sensors, or in AlGaN/GaN high electron mobility transistors [14], characterized by their simultaneous low-frequency noise of gate and drain currents.

2) To explain and predict the character of noise, including reversible and irreversible aspects, the character of frequency-energy dependences, temperature and mechanical changes, it is advisable to involve the mechanism of tensile quasi defects. It seems to be more useful in reducing the size of researched materials and devices to a nanoarea.

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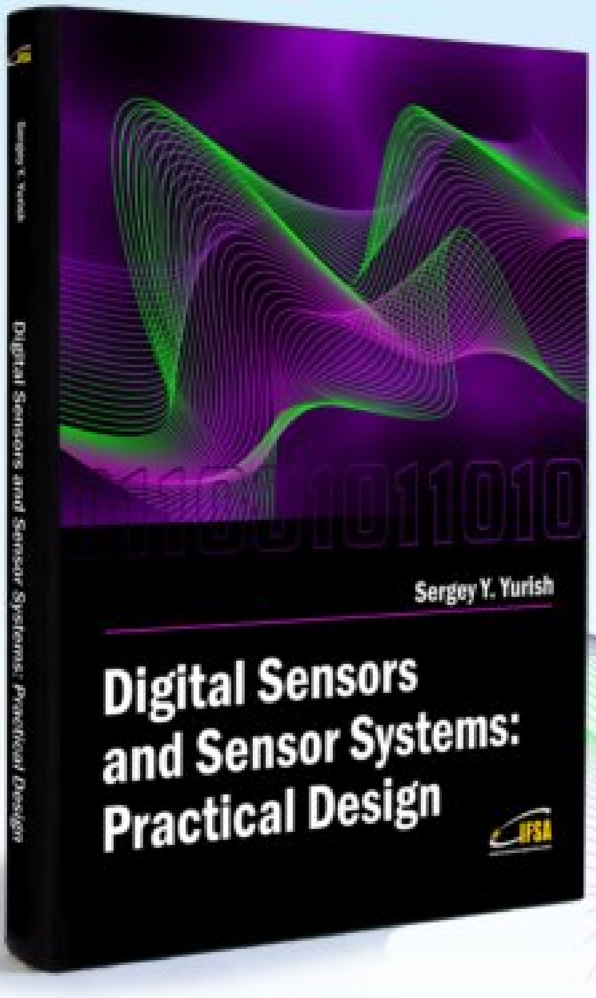
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