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Enhancement of radiation-induced EPR signal in bioapatites

V.V. Nosenko[1,2*], I.P. Vorona[1], S.V. Lemishko[1], I.S. Golovina[1], V.O. Yukhymchuk[1], S.M. Okulov[1], V.B. Neimash[3], V.Yu. Povarchuk[3], S.O. Solopan[4], A.G. Belous[4]

[1]*V. Lashkaryov Institute of Semiconductor Physics, National Academy of Sciences of Ukraine,*
45, prosp. Nauky, 03680 Kyiv, Ukraine
[2]*National University “Kyiv-Mohyla Academy”, 2, Skovorody str., 04070 Kyiv, Ukraine*
[3]*Institute of Physics, National Academy of Sciences of Ukraine, 46, prosp. Nauky, 03680 Kyiv, Ukraine*
[4]*V. Vernadsky Institute of General and Inorganic Chemistry, National Academy of Sciences of Ukraine,*
32/34, Academician Palladin Ave., 03142 Kyiv, Ukraine
[*]*E-mail: vvnosenko@ukr.net*

Abstract. Amplification of a dosimetric EPR signal in mineralized biological materials available in limited quantities has been demonstrated in this paper. Powders of irradiated enamel, dentin, and bone tissue were placed into silica ampoules with the outer diameter close to 1.4 mm. To amplify the signal, the dielectric insert in the form of cylinder with the outer radius 2.85 mm, the inner radius 0.75 mm, and the height 1.85 mm made of a high-κ and low-loss ceramic material $BaTi_4O_9$ + 8.5% ZnO has been used. It has been shown that maximum signal amplification (about an order of magnitude) has been achieved when the sample is completely inserted into this dielectric. It has been found that the line shape of the dosimetric signal is not distorted, if using the dielectric insert. Decomposition of the amplified EPR spectra allowed us to determine the relative contribution of two types of CO_2^- radicals to the dosimetric signal, which coincides with the literature data.

Keywords: EPR, paramagnetic defect; bioapatite, dielectric insert.

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1. Introduction

EPR dosimetry and EPR dating are based on the study of radiation defects formed in various materials under the action of ionizing radiation. It is well known that irradiation of biological tissues, namely: tooth enamel, dentin, bone, *etc.*, with γ- or X-rays induces the so-called dosimetric EPR signal near $g = 2$. The signal intensity linearly depends on irradiation within a wide range of absorbed doses [1, 2], which makes synthetic analogues of biological tissues a promising material for dosimetry. In addition, it was found that the intensity of the radiation-induced EPR signal remains unchanged over a long period of time, *i.e.*, the paramagnetic defects that cause this EPR signal are stable. Their estimated lifetime is longer than 10^{10} years [3, 4]. These properties of the radiation defects led to their use in retrospective EPR dosimetry to determine radiation injuries to people affected by nuclear disasters, such as the nuclear bombing of Hiroshima and Nagasaki, as well as accidents at nuclear power plants in Chernobyl and Fukushima [5–10]. Another close area for practical applications of radiation-induced EPR signals in biological tissues is EPR dating, where it is used to determine the age of fossil finds [11–13].

An extensive study of the properties of the EPR dosimetric signal showed that radiation-induced EPR signals are mainly caused by two types of paramagnetic centers – axial and orthorhombic CO_2^- radicals that are characterized by radio-spectroscopic parameters $g_{||} = 1.9975$, $g_{\perp} = 2.0023$ and $g_x = 2.0030$, $g_y = 2.0015$, $g_z = 1.9975$, respectively [14]. Both radicals are localized in carbonate-containing hydroxyapatite – the main mineral (inorganic) component of biological tissues and differ in the symmetry of the local environment. Analysis of the EPR dosimetric signal line shape allowed determining their relative contribution. It was found that the ratio between axial and orthorhombic radicals depends on several factors: the type of irradiation (UV or γ) [15], thermal treatment of biological tissues [16], storage time of fossil materials [17]. Therefore, analyzing not only the intensity of dosimetric signal but also its shape is an important expansion of the capabilities inherent to EPR dosimetry and EPR dating.

To increase the sensitivity is one of major challenges for these techniques, because EPR signal is rather weak in the dose range of interest for human dosimetry, and it is often impossible to increase the sample amount to enhance the intensity of EPR signal.

For example, to increase the sensitivity, it was proposed to detect the tooth enamel signals at low temperatures and rapid passage conditions, because the signal of second harmonic quadrature absorption is much less saturable and more intense at 77 K [18]. This method gave an improvement in the signal-to-noise ratio four times as compared to the conventional way. It provides the possibility to detect four times smaller absorbed doses, however, along with the opportunity to detect the presence of a weaker signal, information about the line shape of the signal under conditions of fast passage is missed. Another method to increase the EPR sensitivity includes application of over-modulated EPR signals [19]. However, this method also has similar limitations. Therefore, this work is aimed at increasing the sensitivity of EPR techniques without loss of information about the EPR signal line shape. For this purpose, we used a dielectric insert. It is known that dielectric inserts placed into standard cavity of EPR spectrometer concentrate the microwave magnetic field H_1 in the sample region, and so increases the cavity filling factor [20–25], which leads to amplification of the signal from the sample. In this work, we use the dielectric insert made of high-κ material with dielectric constant $\kappa \approx 36$ developed by us earlier [26], as well as we analyze their influence on the intensity and line shape of the dosimetric signal in different mineralized biological tissues.

2. Materials and methods

We used powders of tooth enamel, dentin, and bone tissue prepared using the procedure adopted in retrospective EPR dosimetry [27]. For this purpose, careful separation of the enamel and dentin was carried out using dental instruments. Dentin, enamel, and bone tissue were ground into a powder with a granule size of ~100 µm by using a mortar and pestle. The resulting powders were irradiated at room temperature. In accordance with the measurement technique [27], the EPR spectra were recorded a month after irradiation, which enabled to avoid the influence of EPR signals caused by short-lived radicals on these spectra.

Dielectric insert (DI) was made of ceramic barium tetratitanate ($BaTi_4O_9$ + 8.5% ZnO). Calculation of the DI sizes, simulation of the microwave electromagnetic field distribution in a metallic TE_{102} cavity and studying dielectric insert effect on this distribution were carried out using the Ansys HFSS simulation software. The geometric parameters of DI are as follows: the outer radius 2.85 mm, the inner radius 0.75 mm and the height 1.85 mm. For more details, see [26, 28].

The irradiated powders were placed into the silica tubes with an outer diameter of 1.4 mm. The heights of the enamel, dentin, and bone tissue powders were 1.8, 2, and 3 mm, respectively. Different heights of the studied powders were used to check the effect of the dielectric insert with a full or partial entry of the test sample into it on the intensity and line shape of the EPR dosimetric signal. EPR measurements were carried out at room temperature by using X-band spectrometer Radiopan SE/X2544 (~9.5 GHz) equipped with rectangular TE_{102} metallic cavity.

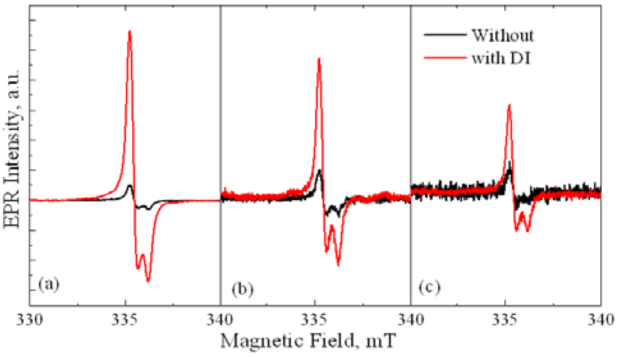

Fig. 1. EPR signal from enamel (a), dentine (b), and bone (c) powders in the rectangular cavity without and with the dielectric insert. (Color online)

3. Results and discussion

Fig. 1 shows the EPR spectra of enamel, dentine, and bone powders measured at identical operation parameters of the EPR spectrometer by using a standard rectangular TE_{102} cavity with and without the insert. It is known that the introduction into the cavity of a sufficient amount of dielectric material with high κ (in our case it is a dielectric insert of a cylindrical shape) leads to a significant shift of its resonance frequency. In our case, this shift was ~ 140 MHz. It means that EPR spectra of the samples, recorded with and without dielectric insert, were observed in different magnetic fields. Since the position and line shape of the dosimetric signal are determined by the electron Zeeman interaction linearly dependent on the external static magnetic field H_0, then for a better visual comparison, the spectra were shifted to the same frequency.

The EPR spectra recorded without dielectric insert are characterized by low intensity, which is caused by the low amount of studied samples. The signal-to-noise ratio in the spectra allows one to analyze the shape of dosimetric signal only in the sample of enamel, while in the sample of bone tissue, even determination of the signal intensity is possible only with a large error. This is caused by the difference in radiation sensitivity of enamel, dentin and bone to γ-irradiation and is in good agreement with the literature data [29, 30]. The usage of DI increases the EPR signal intensity by ~ 11, 8, and 6.5 times for enamel, dentin, and bone powders, respectively. The different gain took place because the used samples were capillaries with different heights of the studied powders. The enamel powder was completely inside the dielectric insert, while the dentin and bone powder were partially outside the dielectric insert. *I.e.*, in dentin and bone samples, the EPR signal was enhanced only by some part of the sample. The EPR spectra recorded using the dielectric insert are characterized by a significantly higher signal-to-noise ratio and allow more accurate determination of their intensity.

Amplification of EPR signals when using DI is due to increasing the filling factor of the cavity and the concentration of the magnetic component H_1 of the

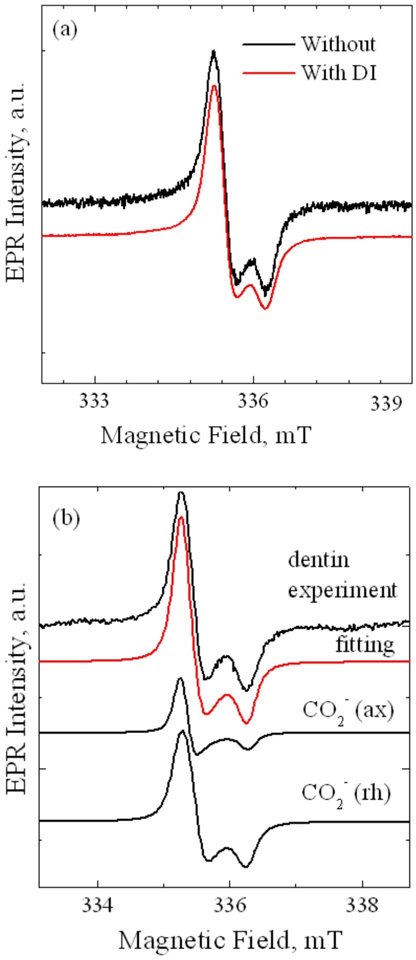

Fig. 2. Normalized EPR dosimetric signals from enamel powder recorded with and without dielectric insert (a). Dentin experimental spectrum recorded with dielectric insert and its modeling (b).

microwave field [26]. The filling factor does not affect the line shape of EPR signal, while an increase in H_1 can lead to saturation effects that significantly distort the line shape of EPR signal. As noted above, the EPR dosimetric signal in mineralized biological tissues is due to two types of CO_2^- radicals: the axial and orthorhombic ones. They are characterized by different relaxation times [16, 31] and at high microwave powers, the dosimetric signal is saturated and, consequently, the line is broadened [32]. Fig. 2a shows the normalized EPR spectra of enamel powder, recorded with and without the dielectric insert. There is no visual distortion of the spectrum line shape. Nevertheless, to check the change in the line shape of EPR spectra of all the samples recorded using DI and also to determine the contributions of axial and orthorhombic centers to the total EPR spectrum, deconvolution procedure was performed. As an example, the results of applying this procedure to the dentine experimental spectrum are shown in Fig. 2b. The radio-spectroscopic parameters for modeling the corresponding components were taken from the literature [14]. As seen from Fig. 2b, the agreement between the experimental and model spectra is rather well. The estimated ratio of axial and orthorhombic CO_2^- radicals in enamel, dentin, and bone was 1:5, which is consistent with that obtained in [16, 30].

4. Conclusions

The obtained results clearly demonstrate the possibility of using a dielectric insert made of high-κ and low-loss material to effectively enhance the dosimetric signal of mineralized biological tissues used in EPR dosimetry and EPR dating. In particular, the dielectric insert made of ceramic barium tetratitanate ($BaTi_4O_9$ + 8.5% ZnO) with $\kappa \approx 36$ and $\tan\delta \approx 1.9 \cdot 10^{-4}$ amplifies the signal by about an order of magnitude, provided that a small amount of test material is used (it must be placed completely into the dielectric insert). The shape of the EPR dosimetric signal is not distorted, which enables to carry out further analysis to separately determine the contribution of the axial and orthorhombic radicals that form the radiation-induced EPR spectrum.

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Authors and CV

Valentyna V. Nosenko, PhD, Senior Researcher at the V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The area of her scientific interests includes EPR and ENDOR spectroscopy, defects in solids. E-mail: vvnosenko@ukr.net; https://orcid.org/0000-0002-9768-1724

Igor P. Vorona, Dr. Sci., Leading researcher at the V. Lashkaryov Institute of Semiconductor Physics, NASU. The area of his scientific interests includes EPR, ODMR and ENDOR spectroscopy, defects in solids. https://orcid.org/0000-0002-4315-1576.
E-mail: ip_vorona@yahoo.com

Serhii V. Lemishko, Junior Researcher at the V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The area of his scientific interests includes EPR spectroscopy, defects in solids.

Iryna S. Golovina, Dr. Sci., Leading researcher at the V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The area of her scientific interests includes EPR spectroscopy, defects in solids, ferroelectrics. https://orcid.org/0000-0003-1199-7339

Volodymyr O. Yukhymchuk, Prof., Dr. Sci., Head of the Department at the V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The area of his scientific interests includes Raman spectroscopy, luminescence, quantum dots, semiconductors, nanostructures.

Serhii M. Okulov, Senior Researcher at the V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The area of his scientific interests includes EPR and ENDOR spectroscopy, defects in solids. https://orcid.org/0000-0001-7217-928X

Volodymyr B. Neimash, Dr. Sci., Head of the Laboratory of radiation technology at the Institute of Physics, NAS of Ukraine. Field of researches: nanomaterials, radiation physics of condensed mater. https://orcid.org/0000-0002-2876-549X.
E-mail: neimash@gmail.com

Vasyl Yu. Povarchuk, PhD, Senior Researcher at the Institute of Physics, NAS of Ukraine. The area of his scientific interests includes radiation physics of condensed mater. https://orcid.org/0000-0002-0868-000X

Serhii O. Solopan, Dr. Sci., Senior Researcher at the V. Vernadsky Institute of General and Inorganic Chemistry, NAS of Ukraine. The area of his scientific interests includes solid state chemistry. https://orcid.org/0000-0001-8079-3626

Anatolii G. Belous, Prof., Dr. Sci., Academician of NAS of Ukraine (Inorganic Chemistry), Head of the Department at the V. Vernadsky Institute of General and Inorganic Chemistry, NAS of Ukraine. https://orcid.org/0000-0001-7808-3828

Authors' contributions

Nosenko V.V.: sample Preparation, experimental study, spectra analysis, writing – original draft.
Vorona I.P.: conceptualization, spectra analysis, writing – review & editing.
Lemishko S.V.: cavity calculation, sample preparation, writing – original draft.
Golovina I.S.: cavity calculation, writing – review & editing.
Yukhimchuk V.O.: resources, writing – review & editing, project administration.
Okulov S.M.: experimental study.
Neimash V.B.: exposure dose assessment, writing – review.
Povarchuk V.Yu.: sample irradiation.
Solopan S.O.: ceramics synthesis, insert manufacturing, writing – review & editing.
Belous A.G.: ceramics synthesis, Insert manufacturing, writing – review & editing, project administration.

Підсилення радіаційно-індукованого сигналу ЕПР у біоапатитах

В.В. Носенко, І.П. Ворона, С.В. Лемішко, І.С. Головіна, В.О. Юхимчук, С.М. Окулов, В.Б. Неймаш, В.Ю. Поварчук, С.О. Солопан, А.Г. Білоус

Анотація. Продемонстровано підсилення дозиметричного сигналу ЕПР у мінералізованих біологічних матеріалах, доступних в обмеженій кількості. Порошки опроміненої емалі, дентину та кісткової тканини вміщувалися у кварцові пробірки зовнішнім діаметром 1,4 мм. Для підсилення сигналу використано діелектричну вставку у вигляді циліндра із зовнішнім радіусом 2,85 мм, внутрішнім радіусом 0,75 мм і висотою 1,85 мм, виготовлену з керамічного матеріалу $BaTi_4O_9$ + 8,5% ZnO з низькими діелектричними втратами. Показано, що максимальне підсилення сигналу (приблизно на порядок) досягається, коли зразок повністю знаходиться всередині діелектричної вставки. Виявлено, що форма лінії дозиметричного сигналу не спотворюється при використанні діелектричної вставки. Розкладання підсилених спектрів ЕПР на компоненти дозволило визначити відносний внесок двох типів радикалів CO_2^- у дозиметричний сигнал, що збігається з літературними даними.

Ключові слова: ЕПР, парамагнітний дефект, біоапатит, діелектрична вставка.