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CAPACITIVE TRANSDUCERS BASED ITO/POLYIMIDE/Al_2O_3 THIN FILM STRUCTURE

L. ZAITSEVA[1*], O. VODORIZ[2], R. ZAITSEV[3]

[1] Department of Natural Science, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, UKRAINE
[2] Department of Physics, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, UKRAINE
[3] Materials for Electronics and Solar Cells Department, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, UKRAINE
[*]e-mail: liliia.zaitseva@khpi.edu.ua

ABSTRACT *It has been developed and approbated prototypes of thin-film capacitor transducers based on Al/ITO/polyimide/Al_2O_3 heterosystem for capacitive acoustic control in metal objects in the modes of simultaneous acoustic signal reception and generation by capacitive transducers and in certain modes of acoustic signal generating or receiving that can realize objects monitoring with sensitivity at the level of piezoelectric transducers. The developed prototype of thin film capacitive transducer for monitoring pipelines by longwave capacitive method allows increasing the maximum distance between the capacitive transducers up to 10 meters. At a substrate temperature of 300 ^oC and a specific power of the magnetron of 0.31 W/cm^2 on Upilex polyimide films were obtained layers of capacitive transducers with a thickness of 0.2-0.3 μm with a surface resistance of 8 Ohm/□, while the concentration of charge carriers was $8{,}3\cdot10^{20}$ cm^{-3} and mobility - 44 cm^2/(V·s). It has been engineered the thin film capacitive transducers which by using the polyamide film with 15 microns thickness of and alumina film with 1 micron thickness allow to increase the sensitivity of such method in 7-8 times, and the additional use of thin crystalline Al_2O_3 films deposited on a substrate of polyimide, allows to increase the value of the dielectric constant of the layer of the capacitive transduser from 3-4 relative units to 8.5-11.5 relative units. Experimental studies of the crystal structure of the developed transducers by X - ray diffractometry and research of their dielectric properties were carried out. The device was tested and the possibility of its use was shown along with the generally accepted methods of defectoscopy. It is shown that using the magnetron sputtering technology, which provides high adhesion to polyimide substrate layers, made possible produce the capacitive transducers for objects with various shape. Proposed and patented: capacitor method for receiving acoustic signals in non-destructive control and transducer of ultrasonic acoustic wave's excitation and receiving.*
Keywords: *thin film, polyimide; ITO; transducer; capacitive method; metal defectoscopy; dielectric constant*

ЄМНІСНИЙ ПЕРЕТВОРЮВАЧ НА ОСНОВІ ТОНКОПЛІВКОВОЇ СТРУКТУРИ ІТО/ПОЛІІМІД/Al_2O_3

Л. В. ЗАЙЦЕВА[1], О. С. ВОДОРІЗ[2], Р. В. ЗАЙЦЕВ[3]

[1] Кафедра природничих наук, Національний технічний університет «Харківський політехнічний інститут», Харків, УКРАЇНА
[2] Кафедра фізики, Національний технічний університет «Харківський політехнічний інститут», Харків, Україна
[3] Кафедра фізичного матеріалознавства для електроніки та геліоенергетики, Національний технічний університет «Харківський політехнічний інститут», Харків, УКРАЇНА

АНОТАЦІЯ *Розроблено та апробовано дослідні зразки тонкоплівкових ємнісних перетворювачів на основі гетеросистеми Al/ITO/поліімід/Al_2O_3 для проведення ємнісного акустичного контролю у металевих виробах у режимах одночасного прийому і генерації акустичного сигналу ємнісними перетворювачами та в окремих режимах генерації або прийому акустичного сигналу, які дають можливість реалізувати контроль об'єктів з чутливістю на рівні п'єзоелектричних перетворювачів. Розроблено дослідний зразок тонкоплівкового ємнісного перетворювача для проведення контролю трубопроводів довгохвильовим ємнісним методом, що дає змогу збільшити максимальну відстань між ємнісними перетворювачами при контролі до 10 метрів. При температурі підкладки 300 ^oC та питомій потужності магнетрону 0,31 Вт/см^2 на поліімідних плівках фірми Upilex були отримані шари ємнісних перетворювачів завтовшки 0,2-0,3 мкм з поверхневим електроопором 8 Ом/□, при цьому концентрація носіїв заряду становила $8{,}3\cdot10^{20}$ см$^{-3}$, рухливість - 44 см^2/(В·с). Створені тонкоплівкові ємнісні перетворювачі, які дають змогу за рахунок використання поліімідної плівки товщиною 15 мкм та плівки оксиду алюмінію товщиною 1 мкм збільшити чутливість методу у 7-8 разів, а додаткове використання тонких кристалічних плівок Al_2O_3, осаджених на підкладку з поліїміду, дозволяє підвищити величину діелектричної проникності прошарку ємнісного перетворювача від 3-4 відн.од., характерних для поліміду, до 8,5-11,5 відн. од. Проведено експериментальні дослідження кристалічної структури розроблених перетворювачів методом рентгендифрактометрії та дослідження їх діелектричних властивостей. Проведено апробацію приладу та показано можливість його використання поряд із загальноприйнятими методами дефектоскопії. Показано, що використання технології магнетронного розпилення, котра забезпечує високу адгезію шарів до поліімідної підкладки, дає змогу одержувати ємнісні*

перетворювачі для виробів різноманітної форми. Запропоновані і запатентовані: конденсаторний спосіб прийому акустичних сигналів при неруйнівному контролі та перетворювач збудження і прийому ультразвукових акустичних хвиль.
Ключові слова: *тонкі плівки; поліімід; ІТО; перетворювач; ємнісний метод; дефектоскопія металів; діелектрична стала*

Introduction

The need to simplify the control technology of metal products macrodefects in terms of industrial production has led to the further development of widely used acoustic methods [1]. They are based on piezoelectric devices that implement the acoustic method using special fluids to ensure the necessary acoustic contact. Considerable experience in the practical use of this method has identified areas in which it isn't effective [2,3]. So it is impossible to use liquid for acoustic control of the products with the polluted surface struck by corrosion, or with coverings (paint, polymeric films and other insulating coverings), hot and cold products. Thus, for the needs of practical defectoscopy it is necessary to create devices that will allow to conduct liquid-free acoustic control [4,5].

Formulation of a problem

Promising among the devices of liquid-free acoustic control can be means created on the basis of capacitive method of generation and reception of acoustic signals, which has a fundamentally different physical mechanism of acoustic signal generation in the control object, its surface is one of the plates of the capacitive composition and itself generates a signal without the need to use liquid to ensure acoustic contact. However, the existing capacitive compositions don't allow to obtain the necessary sensitivity of the method and the task is to find, create and study the latest capacitive transducers based on thin film layers, among which are very promising layers of polyimide, ITO [6,7] (Indium Tin Oxide, mixed indium oxide and tin $(In_2O_3)_{0,9}$-$(SnO_2)_{0,1}$) and Al_2O_3 alumina.

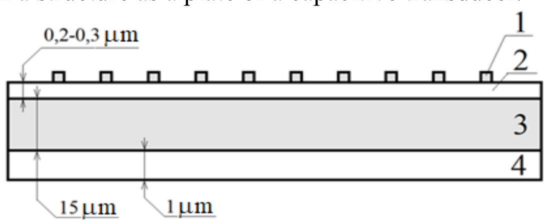
Methods of obtaining samples

The following key requirements for increasing the sensitivity and efficiency of capacitive transducers follow from the practice of using classical capacitive transducers for defectoscopy by the capacitive method, which are as follows:
- reducing the dielectric layer thickness;
- increasing the dielectric constant value of the layer.

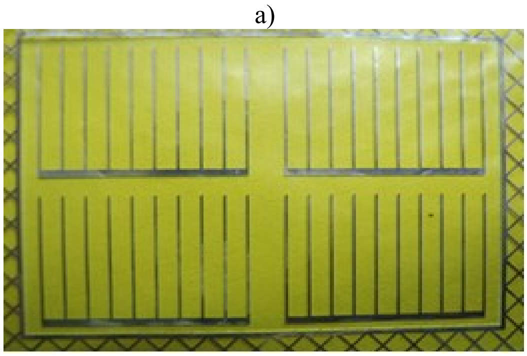
From the view point of the requirements fulfillment, the possibility of using a polyamide film with a thickness of 15 to 125 μm as a dielectric layer and a base for capacitive transducers, which is 10 times less than the thickness of classical dielectric layers, is quite relevant. When creating a capacitive converter with the Al/ITO/polyimide/Al_2O_3 structure, a polyimide of the Upilex-S brand with a thickness of 15 μm was used, the surface of which was previously cleaned. On one of the polyimide sides by the method of non-reactive magnetron sputtering on direct current on VUP-5M vacuum plant in the following technological conditions the ITO layer was obtained: the discharge gap length - 70 mm; deposition time - 30 minutes; the initial residual pressure in the vacuum chamber was $3 \cdot 10^{-7}$ Pa and the working pressure in the process of target spraying - $1.5 \cdot 10^{-4}$ Pa; ITO target consisted of 90 Wt % In_2O_3 and 10 Wt % SnO_2 and pressed under a pressure of approximately 12 kg/cm^2; the substrate temperature was 300 ^oC, the magnetron specific power was 0.28 W/cm^2. Current-conducting tracks made of aluminum on the ITO layer surface was created by the method of thermal resistive spraying on a VUP-4 vacuum unit in the following technological conditions: spraying was carried out through a suitable mask at a vacuum of $2 \cdot 10^{-5}$ Pa, the substrate was heated to 110 ^oC, spraying time with tungsten evaporators was 120 s. On the other side of the polyimide film in similar technological conditions to the ITO layer was obtained a Al_2O_3 layer [8]. A schematic cross-sectional view of such a device is shown in Figure 1, a. According to the above technology, a prototype of a thin-film capacitive transducer for acoustic control of metal products was created, the physical configuration of which is shown in Figure 1, b.

Study of the crystal structure of thin-film capacitive transducers

To achieve the maximum quality of such structures it is necessary to control the parameters of the ITO layer: its crystal structure, which determines most of its properties, including mechanical, and its dependence on deposition conditions, as well as its electrical resistance, the values of which determine the possibility of using such a structure as a plate of a capacitive transducer.



a)



b)

Fig. 1 - A schematic cross-sectional view (a) and the physical configuration (b) the proposed thin-film capacitive transducer: 1 – aluminum current-conducting tracks; 2 - ITO layer with a thickness of 0.2 – 0.3 μm; 3 - polyimide film with a thickness of 15 μm; 4 - Al_2O_3 layer with a thickness of 1 μm

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Studies of the film layers crystal structure were carried out by the traditional X-ray diffraction method [9] on DRON-4 X-ray machine with automatic recording of the diffraction spectrum using a computer with continuous 2θ-scanning in the range of angles 2θ = 20 – 75 with Bragg-Brentano focusing (θ-2θ) in cobalt anode radiation. The surfaces of ITO and Al_2O_3 layers were also studied using a REM-100U scanning electron microscope. The obtained X-ray diffraction patterns and surface micrographs are shown in Fig. 2 and 3, for the ITO layer and the Al_2O_3 layer, respectively.

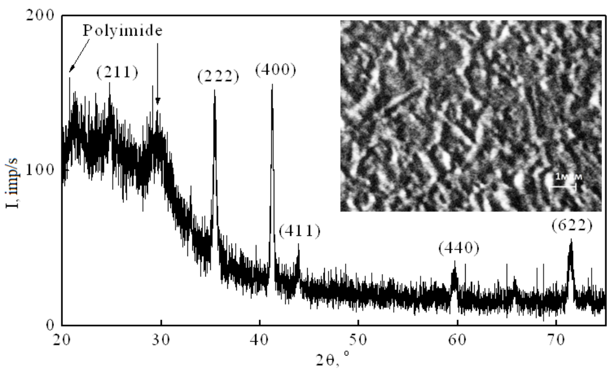

Fig. 2 - X-ray diffractogram and micrograph of the ITO layer surface deposited on the polyimide film

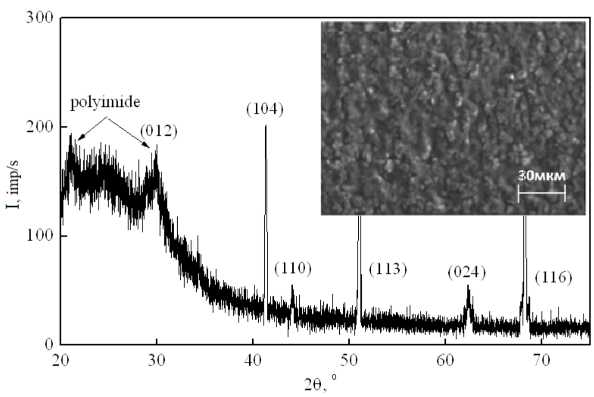

Fig. 3 - X-ray diffractogram and micrograph of the Al_2O_3 layer surface deposited on the polyimide film

Analysis of X-ray diffractograms of ITO layers obtained by the nonreactive magnetron sputtering method (Fig. 2) showed that all layers have a crystal structure of In_2O_3stable cubic modification. The latter is unequivocally evidenced by the presence of reflections from the planes (221), (222), (400), (411), (332), (431), (440), (611), (622). The results of the total integral intensity ratio calculations of all peaks observed on the diffraction pattern to the thickness of the ITO layer indicate the presence of a X-ray amorphous phase small amount in the samples. It was also experimentally found that at a substrate temperature of 300 °C, the film growth occurs with a predominant orientation in the direction <111>.

The structure analysis of the Al_2O_3 layer showed that all layers have a crystal structure of a stable rhombohedral modification of α-Al_2O_3 with lattice parameters a = 4.759 Å, c = 12.993 Å. This is clearly evidenced by the presence of reflections from the planes (012), (104), (110), (113), (024) and (116) [10]. The X-ray diffractogram analysis shows that the Al_2O_3 layer has a stable crystal structure, and as a consequence, has stable electrical parameters that correspond to the structure.

Study of the crystal structure of thin-film capacitive transducers

As already mentioned, for the manufacture of thin-film capacitive converters based on the ITO/polyimide/Al_2O_3 structure along with the layers crystal structure, it is also necessary to control the surface electrical resistance of the conductive layer, the values of which determine the possibility of using such a structure as a plate of the capacitive transducer without considerable losses of a useful signal, and the dielectric constant of the dielectric layer, which significantly affects the useful signal magnitude.

To control the surface electrical resistance of the ITO layer, the four-probe method was used [11], and the surface electrical resistance ($R_\square$) of the ITO layers determined by this method is equal to 8-15 Ω/m. E.m.f. Hall studies indicate that the obtained resistivity value is due to the main charge carriers concentration from about $8.3\cdot10^{20}$ cm^{-3} and the main charge carriers mobility at the level of 44 $cm^2/(V\cdot s)$.

Studies of the polyamide film dielectric constant and the polyimide/Al_2O_3 structure were performed for the excitation signals frequencies in the range of 10 - 10^7 Hz, which were generated by a GSS-20 signal generator type. The capacitance of the capacitor structure was measured using an RLC-meter type E318, and the data are shown in Fig. 4.

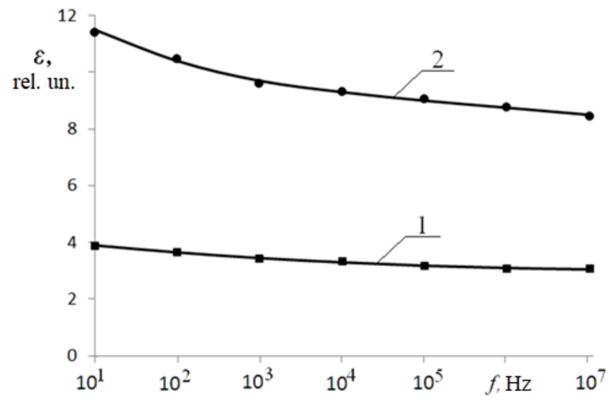

Fig. 4 - Dependence of polyimide film dielectric constant (1) and polyimide/Al_2O_3 structure of (2) on frequency of excitation signal

The study results show an increase in the dielectric constant of the polyimide/Al_2O_3 structure, which is 8.5 – 11.5 rel. unit, relative to the polyimide film (3 – 3.5 rel. unit) approximately 3 times at frequencies of the excitation signal in the range of 10 Hz – 10 MHz [12]. This circumstance confirms the assumption about the possibility of increasing the layer dielectric constant by applying a Al_2O_3 thin layer on the polyimide film. A 3-fold increase in the dielectric constant will lead to an additional 3-fold increase in the sensitivity of the capacitive transducer based on such a layer.

Approbation of the device

To confirm the possibility of method sensitivity increasing due to the use of a capacitive transducer based on the Al/ITO/polyimide/Al_2O_3 structure in comparison with classical transducers, a series of samples from aluminum was studied at an oscillation frequency of 2.5 MHz The received signals obtained oscillograms the for transducers both types with the same magnitude of the excitation signal are shown in Fig. 5.

As can be seen from Fig. 5, the received signal value in the case of using a thin-film transducer increases by 7.6 times compared to the classical transducer, which correlates well with the dielectric constant dielectric layers measurements and taking into account thickness reduction of the dielectric layer. Thus, the increase in the capacitive method sensitivity in the case of the thin-film capacitive transducers use based on the Al/ITO/polyimide/Al_2O_3 structure is experimentally confirmed.

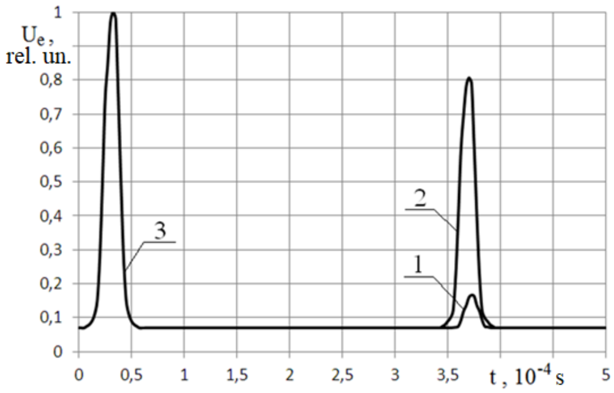

Fig. 5 - Normalized oscillograms of the received signals from the classical capacitive transducer (1) and the proposed thin-film based on the Al/ITO/polyimide/Al_2O_3 structure (2) in comparison with the same excitation signal (3)

Conclusions

It is proposed to use as a dielectric layer in defectoscopy of metal products by capacitive method a thin polyimide film, the thickness of which is two orders of magnitude less than the thickness of classical dielectric layers, and is 15 μm, and the dielectric constant is 3-4 rel. units, which allows, respectively, to increase the sensitivity of the capacitive method by about 100 times. ITO layers with a thickness of 0.2 – 0.3 μm (surface resistance was 8 – 15 Ω/m, the charge carriers concentration was $8.3\cdot10^{20}$ cm^{-3}, mobility was 44 $cm^2/(V\cdot s)$) were obtained on polyimide films from Upilex at a substrate temperature of 300 ºC and a magnetron specific power of 0.31 W/cm^2. It was found that the additional use of Al_2O_3 thin crystalline films deposited on a polyimide substrate, allows to increase the dielectric constant of the capacitive transducer layer from 3-4 rel. unit characteristic of polyimide, up to 8.5 – 11.5 rel. unit. The obtained growth in the dielectric constant value allows to increase the capacitive method sensitivity by at least 3 times. A prototype of a thin-film capacitive transducer for acoustic control of metal products based on the Al/ITO/polyimide/Al_2O_3 structure was created. An experimental study of the prototype was performed and it was found that the use of a thin-film capacitive transducer based on the Al/ITO/polyimide/Al_2O_3 structure increases the sensitivity of the capacitive method by 7.6 times.

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Відомості про авторів (About authors)

Зайцева Лілія Василівна – кандидат технічних наук, доцент кафедри природничих наук, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: 0000-0002-0668-711X; e-mail: liliia.zaitseva@khpi.edu.ua.

Liliia Zaitseva – Ph. D., Associate Professor, Department of Natural Science, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, Ukraine; ORCID: 0000-0002-0668-711X; e-mail: liliia.zaitseva@khpi.edu.ua.

Водоріз Ольга Станіславівна – кандидат фізико-математичних наук, кафедра фізики, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: 0000-0002-2951-4887; e-mail: olga.vodorez@gmail.com.

Olga Vodoriz – Ph. D., Senior Lecturer, Department of Physics, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, Ukraine; ORCID: 0000-0002-2951-4887; e-mail: olga.vodorez@gmail.com.

Зайцев Роман Валентинович– доктор технічних наук, доцент, завідувач кафедри фізичного матеріалознавства для електроніки та геліоенергетики, Національний технічний університет «Харківський політехнічний інститут»; м. Харків, Україна; ORCID: 0000-0003-2286-8452; e-mail: roman.zaitsev@khpi.edu.ua.

Roman Zaitsev – D.Sc., Docent, Head of Department, Materials for Electronics and Solar Cells Department, National Technical University "Kharkiv Polytechnic Institute", Kharkiv, Ukraine; ORCID: 0000-0002-2951-4887; e-mail: roman.zaitsev@khpi.edu.ua.

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АННОТАЦИЯ *Разработаны и апробированы опытные образцы тонкопленочных емкостных преобразователей на основе гетеросистемы Al/ITO/полиимид/Al_2O_3 для проведения емкостного акустического контроля в металлических изделиях в режимах одновременного приема и генерации акустического сигнала емкостными преобразователями и в отдельных режимах генерации или приема акустического сигнала, которые позволяют реализовать контроль объектов с чувствительностью на уровне пьезоэлектрических преобразователей. Разработан опытный образец тонкопленочного емкостного преобразователя для проведения контроля трубопроводов длинноволновым емкостным методом, что позволяет увеличить максимальное расстояние между емкостными преобразователями при контроле до 10 метров. При температуре подложки 300 °С и удельной мощности магнетрона 0,31 Вт/см^2 на полиимидной пленке Upilex были получены слои емкостных преобразователей толщиной 0,2-0,3 мкм с поверхностным электросопротивлением 8 Ом/□, при этом концентрация носителей заряда составляла 8,3·10^{20} см$^{-3}$, подвижность - 44 см^2/(В·с). Созданы тонкопленочные емкостные преобразователи, которые позволяют за счет использования полиимидной пленки толщиной 15 мкм и пленки оксида алюминия толщиной 1 мкм увеличить чувствительность метода в 7-8 раз, а дополнительное использование тонких кристаллических пленок Al_2O_3, осажденных на подложку из полиимида, позволяет повысить величину диэлектрической проницаемости прослойки емкостного преобразователя от 3-4 отн. ед., характерных для полиимида, до 8,5-11,5 отн. ед. Проведены экспериментальные исследования кристаллической структуры разработанных преобразователей методом рентгендифрактометрии и исследованы их диэлектрические свойства. Проведена апробация прибора и показана возможность его использования наряду с общепринятыми методами дефектоскопии. Показано, что использование технологии магнетронного распыления, которая обеспечивает высокую адгезию слоев к полиимидной подложке, позволяет получать емкостные преобразователи для изделий различной формы. Предложены и запатентованы: конденсаторный способ приема акустических сигналов при неразрушающему контролю и преобразователь возбуждения и приема ультразвуковых акустических волн.*

Ключевые слова: *тонкие пленки; полиимид; ITO; преобразователь; емкостной метод; дефектоскопия металлов; диэлектрическая постоянная*

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