Tom 9 № 5, 05005(4cc) (2017)

Structural-phase and Electrophysical Properties of Nanocomposits Based on the "Glass-Ni₃B" System, Received by the Cathode Beam Annealing

Ya.I. Lepikh*, T.I. Lavrenova, N.M. Sadovaja

Interdepartmental Scientific-Educational Physicotechnical Centre of MES and NAS of Ukraine at the Odessa I.I. Mechnikov National University, 2, Dvoryanskaya str., 06582 Odesa, Ukraine

(Received 24 August 2017; published online 16 October 2017)

In this work the opportunity of replacement of conductors based on the precious ruthenium, silver-palladium and gold materials in thick-film elements of hybrid integrated circuits (HIS) by conductors based on nickel boride has been investigated. The structural-phase and electro-physical properties of the film conducting elements based on Ni_3B , received at annealing with the help of the cathode beam (CB) and in the furnace has been investigated. It has been established, that the functional materials based on Ni_3B are stable enough at radiative-thermal influence and can be used at conducting pastes manufacturing. Conducting elements based on nickel, annealed with the help of CB, on the electrophysical parameters do not concede to conductors based on the ruthenium, silver – palladium compounds, withstand a repeated burning out without the property deterioration.

Keywords: Materials for film elements of hybrid integrated circuits, Structural-phase and physical properties.

DOI: 10.21272/jnep.9(5).05005 PACS number: 60.61

1. INTRODACTION

Nanocomposition materials are widely used in microelectronics, in particular, as film resistive and conducting elements of hybrid integrated circuits (HIC), solar batteries and sensors [1-3]. As a current-carrying phase mainly precious materials are used: ruthenium compounds, silver, palladium and gold. In the given work the opportunity of precious materials replacement by conductors based on nickel boride (Ni $_3$ B), received with the help of an cathode beam has been investigated

2. SAMPLES AND INVESTIGATION METHODS

Thermal processing of nanocomposition was carried out in the furnace and with the help of the cathode beam on electron accelerators of the MULE-6, the MULE-8 type under normal conditions on air.

As an initial material for paste compositions it was used Ni_3B , received by a method of direct synthesis from nickel and boron according to reaction:

$$3Ni + B = Ni_3B$$

in the vacuum furnace of C-1.2,5/25 I1 type. For synthesis it was used carbonyl and an amorphous boron (marks-94). After synthesis the material was grinded on planetary spherical mill in a chalcedonic drum to dispersion of $1900~\rm cm^2/g$. As a constructive material BK-94-1 ceramics and glass C 279-2 were used, and as organic binding - terpineol. Investigations of the material phase structure were carried out by methods of X-ray analysis on the installation URS-01 and diffractometer DRON-3. Identification of the found lines was carried out on card-file ASTM.

3. DETAILS OF THE EXPERIMENT

For the analysis of the change of the initial functional material phase structure (glass, a current-carrying phase) under the action of the thermal and radiation-thermal processing there were manufactured samples as a entire covering by thickness of 200 microns on ceramics BK-94-1. The phase structure of a glass in an initial condition has been established: ${\rm SiO_2} + 1\%~\alpha\text{-SiO_2}$. The phase structure of conducting phase without processing: Ni₃B+1%Ni, a crystal lattice - texture-oriented Ni. The results of investigations had shown, that the phase structure of a glass C 279-2 after radiation-thermal processing in the interval of temperatures 390-950 °C does not change.

The results of conducting composition phase structure change at processing by the cathode beam and in the furnace are given a in Table 1.

For the investigation of the influence of technological modes at processing by the cathode beam of current layer on electrophysical parameters the composition of the following structure has been used: $87\% - Ni_3B$, 10% - C 279-2. Dependences of conductor specific surface resistance on modes of the cathode beam processing are submitted on Fig. 1a, b, c.

Simultaneously with the ρ_s change, the size of adhesion of the conducting elements based on Ni₃B to a surface of a ceramic substrate has been determined. The adhesion in the interval of technological modes (850-950° C) makes not less than 150 kg/cm² for the samples received by the CB annealing. The adhesion in the interval of technological modes (850-950° C) for the samples received by the annealing in the furnace is much below and makes 4-10 kg/cm².

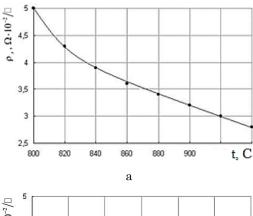
The conducting elements during the HIC manufacturing pass some cycles of the heat treatment and in

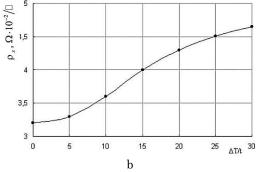
-

^{*} ndl lepikh@onu.edu.ua

Table 1- The phase structure of conducting composition based on Ni_3B changes at processing by the cathode beam and in the furnace

Processing kind	Annealing temperature	Phase structure	Crystal lattice
Processing by CB	390° C	Ni ₃ B+7% Ni+Ni ₄ B ₃	$\alpha = 3.524 \text{ Å}$
	450° C	Ni₃B+13% Ni	-"-
	680° C	Ni ₃ B+21% Ni	-"-
	850° C	$Ni_3B+7\% Ni+\alpha Al_2O_3$	_"-
	900 C	$Ni_4B_3+Ni+\alpha Al_2O_3$	_"-
	950 C	NiB+Ni+α Al ₂ O ₃	-"-
Processing in the furnace	390 C	Ni ₃ B+Ni+H ₃ BO ₃	Texture-oriented Ni
	450 C	Ni ₃ B+Ni+H ₃ BO ₃	-"-
	680 C	Ni _x B _y +Ni	-"-
	850 C	$Ni_{(4-x)}B_{(3+y)}+Ni+NiO$	Ni_4B_3
	900 C	Ni ₄ B ₃ +Ni+NiO	-"-
	950 C	Ni ₄ B ₃ +Ni+NiO	-"-





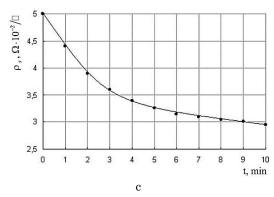


Fig. 1 – Dependences of the conductor specific surface resistance ρ_s on the modes of processing by the cathode beam: annealing temperature (a), temperature speed rise up to $T=850^{\circ}$ C (b), time of the sample treatment at $T=850^{\circ}$ C (c)

addition, as a rule, maintain 3-4 multiple annealing without the change of the properties.

Dependence of the specific surface resistance of the

conducting elements based on Ni_3B at repeated modes of processing by an cathode beam (duration of each cycle 40 c), is given in Fig. 2.

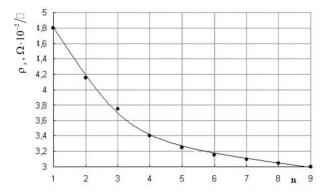


Fig. 2 – Dependence of the specific surface resistance of conductors based on Ni_3B on the amount of processing by the cathode beam

At the forced annealing modes of the conducting elements based on the nickel with the help of CB it is observed that decrease of the specific surface resistance with the annealing temperature increase. The increase of maintain at optimum temperature or decrease of its speed rise has the essentially identical character.

At heating these changes, naturally, are connected to the covering structural transformations, increase of its density, hence the area of contacts between the particles of the functional material. It proves to be true by the investigations of the structure of the conducting elements, received at different modes of processing (Fig. 3).

The occurrence in the conducting layer of a phase αAl_2O_3 does not result in increase of specific surface resistance. This component, probably, takes part in the formation of a transitive layer conductor-ceramics and is responsible for the adhesion, but not for the conductivity of the thick-film structure. It is necessary to note the change of the nickel texture-oriented lattices in the initial material at radiationally-thermal processing to classical, that does its durable relative to action of a different sort of external factors. As the results of microscopic and microprobe analysis of sections of the conducting layer have shown the transition region is more developed and more saturate with nickel at the

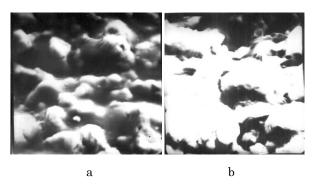


Fig. 3 – The structure of the conducting covering (\Box x5000): (a) $T_{an}=820^{\circ}$ C, t=40 s;(b) $T_{an}=880^{\circ}$ C, t=40 s

annealing with the help of CB as against the samples fabricate on the traditional technology. Besides, the results of microscopic and microprobe analysis of the sections of the samples received on traditional technology have shown the presence of emptiness on the interface of ceramic base sheet - film, in the size from 2 up to 60 microns. It determines the significant decrease of adhesion and, probably, is connected to intensive gas evolution process at annealing.

The diffuse spread of the edges of the topological figure of the conducting paths imposes the restrictions on the resolution of the thick-film technology. At traditional technology of the heat treatment, the migration of a conducting material is high enough and depends on the compound of the thick-film composition and temperature annealing modes. In the given work on the results of the microprobe analysis of the films, annealed by the CB, it is established, that nickel diffuse spread in the time 40-60 s makes approximately 5-7 microns, that allows to densify essentially topology

elements without danger of short circuit between the paths at the expense of diffuse processes so to increase HIS integration degree.

4. CONCLUSIONS

- It has been shown the opportunity of replacement of conductors based on the precious ruthenium, silverpalladium and gold materials in HIS thick-film elements by conductors based on nickel boride.
- It is established, that functional materials based on nickel have stable enough electrophysical parameters at radiationally-thermal processing and can be used at manufacturing of conducting pastes for film resistive, conducting elements of HIS, solar batteries and sensors.
- 3. Conducting elements based on nickel, annealed with the help of CB, on the electrophysical parameters do not concede to conductors based on the ruthenium compound, silver palladium, maintaining repeated annealing without deterioration of their properties. At the forced annealing modes of conducting elements based on nickel with the help of CB the decrease of the specific surface resistance has essentially identical character with the annealing temperature and the maintain at optimum temperature increase, or decrease of its speed rise.
- 4. The amount of adhesion of conducting elements based on Ni₃B to a surface of a ceramic substrate in the interval of CB technological annealing modes (850-950° C) makes not less than 150 kg/cm², that is much higher than at annealing in the furnace (4-10 kg/cm²).
- Diffuse spread of a film conductor at CB thermo treatment allows significantly condense elements of topology without the danger of short circuit between paths and hence to increase the HIS integration degree.

Структурно-фазовые и электрофизические свойства нанокомпозитов на базе системы «стекло-Ni₃B», полученные отжигом электронным пучком

Я.И. Лепих, Т.И. Лавренова, Н.М. Садовая

Межведомственный научно-учебный физико-технический центр МОН и НАН Украины при Одесском национальном университете имени И.И. Мечникова, ул. Дворянская, 2, 06582 Одесса, Украина

В работе исследована возможность замены проводников на основе драгоценных рутениевых, серебро — палладиевых и золотых материалов в толстопленочных элементах гибридных интегральных схем проводниками на основе борида никеля. Исследовано структурно-фазовые и электрофизические свойства пленочных проводниковых элементов на основе Ni₃B, полученных при отжиге с помощью электронного пучка (ЭП) и в печи. Установлено, что функциональные материалы на основе Ni₃B достаточно стабильные при радиационно-термическом воздействии и могут быть использованы при изготовлении проводящих паст. Проводниковые элементы на основе никеля, которые отожжены с помощью ЭП, по своим электрофизическим параметрам не уступают проводникам на основе соединений рутения, серебра-палладия, выдерживая многократный отжиг без ухудшения свойств.

Ключевые слова: Материалы для пленочных элементов гибридных интегральных схем, Структурнофазовые и электрофизические свойства.

Структурно-фазові та електрофізичні властивості нанокомпозитів на базі системи «скло -Ni₃B», отриманих відпалюваннямям електронним пучком

Я.І. Лепіх, Т.І. Лавренова, Н.М. Садова

Міжвідомчий науково-навчальний фізико-технічний центр МОН і НАН України при Одеському національному університеті імені І.І. Мечникова, вул. Дворянська, 2, 06582 Одеса, Україна

В роботі досліджено можливість заміни провідників на основі дорогоцінних рутенієвих, срібло — паладієвих та золотих матеріалів в товстоплівкових елементах гібридних інтегральних схем провідниками на основі бориду нікелю. Досліджено структурно-фазові та електрофізичні властивості плівкових провідникових елементів на основі Ni₃B, отриманих при відпалюванні за допомогою електронного пучка (ЕП) і в печі. Встановлено, що функціональні матеріали на основі Ni₃B достатньо стабільні при радіаційно-термічній дії і можуть бути використані при виготовленні провідникових паст. Провідникові елементи на основі нікелю, які відпалені за допомогою ЕП, за своїми електрофізичними параметрами не поступаються провідникам на основі сполук рутенію, срібла-паладію, витримуючи багатократне випалення без погіршення властивостей.

Ключові слова: Матеріали для плівкових елементів гібридних інтегральних схем, Структурнофазові та електрофізичні властивості.

REFERENCES

- 1. Ya.I. Lepikh, T.I. Lavrenova, Radioelectronics and Inf. Communications, 259 (Київ: 2016).
- В. Недорезов, С. Подшибякин, Электроника No 6, 25 (2011) (V. Nedorezov, S. Podshibyakin, Elektronika No 6,
- 25 (2011)).
- Ya.I. Lepikh, T.I. Lavrenova T.N. Bugayova, N.P. Zatovskaya, P.O. Snigur, Funct. Mater. 21, 297 (2014).