

IMPROVEMENT OF SOLDER FOR JOINING DIFFERENT MATERIALS

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The object of the study was diffusion-hardening gallium solders and other composite components. The market is experiencing an active growth in demand for low-temperature assembly technologies, especially in the field of IoT, wearables, and microsensors. The growing interest in low-temperature solders is due to the fact that they have a very low melting point ($\approx 29.8^\circ\text{C}$), which makes them promising for heat-sensitive electronic components, flexible electronics, 3D assembly without high heating. However, there are problems with their use that need to be solved: gallium corrosion to many metals, surface oxidation leading to poor wettability, slow curing at room temperatures. An experimental study of the phase formation during the interaction of Fe, Ni and iron-nickel alloys 36H and 50H with liquid gallium and tin was conducted. During the research, it was found that the intermetallics that are formed do not always grow according to a parabolic dependence in the coordinates: the thickness of the diffusion layer and the contact time. After calculating the reactive diffusion parameters, it was found that the effective reactive diffusion coefficients during gallium contact in the Fe-Ga, Ni-Ga systems are higher than in the Fe-Sn, Ni-Sn systems. It was found that during the interaction of iron-nickel alloys with liquid gallium, the rate of formation of new phases can be regulated. Iron-nickel alloys with a high nickel content in gallium solder pastes can be used both as solidification accelerators and as stabilizers. The results obtained allow us to state that functional solders are one of the directions of modern microelectronics.

INTRODUCTION

Gallium solder pastes allow to obtain dissimilar connections at room temperature, provide electrical conductivity, vacuum density, as well as the necessary set of mechanical properties and operation of the obtained connections at high temperatures. Gallium pastes are currently used in power electronics, thermal interface materials, micro sensors and MEMS, additive technologies (direct writing, liquid metal printing). Therefore, the study of gallium solders with Fe-Ni fillers is a modern, promising and relevant direction.

ANALYSIS OF LITERARY DATA AND PROBLEM STATEMENT

The paper [1] is devoted to a review of the concept of “magnetic liquid metals” (MLM): methods of preparation of Ga-based liquid metals with ferromagnetic particles, physical properties, examples of applications (locomotion, patterning, soft robots). The authors show the influence of Fe/Ni fillers on the magnetic and rheological properties of pastes.

In [2], the combination of liquid Ga phase with solid particles and polymer matrices is presented, the properties, encapsulation and printing methods are described. This allows the creation of pastes with a solid Fe/Ni filler.

In [3], an experimental study of Ga interactions with practical metallizations (Cu/Ni/Pd, Cu/Ni/Au) was conducted. The mechanisms of Ni-barrier consumption, the formation of intermetallic phases and their influence on the reliability of soldering at low temperatures were shown. In [4], an example of combining Ga with carbon fillers for 3D printing of conductive composites is

provided, the rheology and processability are described. The methodology can be adapted for metal fillers (Fe/Ni). In [5], the development of pasty Ga composites with a high particle loading (Ga_2O_3) to prevent leakage is discussed. It provides important conclusions regarding the preservation of the pasty form and thermal conductivity. In [6], the authors considered the study of Ga interactions with transition metal surfaces at the level of atomic processes. This provides an understanding of the mechanisms of wetting and the formation of interface phases when using Fe/Ni fillers.

To date, the scientific and technical literature has not formulated any substantiated recommendations for reducing the curing time, extending the curing interval, and improving performance characteristics, and this precisely corresponds to the main modern scientific challenges [7, 10, 13].

PURPOSE AND OBJECTIVES OF THE STUDY

The purpose of these studies is to reduce the curing time of solder pastes, expand the curing temperature range, and improve operational characteristics. This will make it possible to provide specific recommendations in the context of low-temperature assembly, development of functional solders, controlled pastes for microelectronics, improvement of mechanical properties of Ga-solders

To achieve the goal, the following tasks were set:

- to perform a set of studies on the selection of iron-nickel alloys as fillers for gallium solder pastes;
- to determine the parameters and products of interaction.

MATERIALS AND RESEARCH METHODS

The object of research is diffusion-hardening gallium solders and other composite components. The subject of research is the physicochemical mechanisms of diffusion hardening of gallium solders and the influence of composite additives on the structure, mechanical and operational properties of the compounds.

The hypothesis of the study is that adding certain compositional components to gallium solders will allow controlling diffusion hardening processes, reducing the brittleness of interphase layers, and increasing the mechanical strength and stability of joints [8, 11, 12].

The assumption is that diffusion hardening occurs mainly due to the mutual diffusion of gallium and the base metal with the formation of intermetallics.

Simplification – the microstructure of the interfacial layers is considered to be homogeneous within each individual phase, without taking into account local fluctuations in chemical composition. The effects of oxidation, surface contamination or microcracks are considered insignificant or controlled.

When conducting the study, experimental physicochemical methods (microhardness method), energy dispersive microanalysis method (the elemental composition of the starting materials and diffusion zones was determined) were used [9].

An experimental study of the phase formation during the interaction of Fe, Ni and iron-nickel alloys 36H and 50H with liquid gallium and tin was conducted. During the research, it was found that the intermetallics that are formed do not always grow according to a parabolic dependence in the coordinates: the thickness of the diffusion layer and the contact time. After calculating the reactive diffusion parameters, it was found that the effective reactive diffusion coefficients during gallium contact in the Fe-Ga, Ni-Ga systems are higher than in the Fe-Sn, Ni-Sn systems. It was found that during the interaction of iron-nickel alloys with liquid gallium, the rate of formation of new phases can be regulated. Iron-nickel alloys with a high nickel content in gallium solder pastes can be used as solidification accelerators and as stabilizers.

RESEARCH RESULTS

1. STARTING MATERIALS AND SAMPLES

The following components were used to manufacture experimental samples: gallium (GL-0), tin (OVCh-000), iron, nickel, iron-nickel alloy (36N), iron-nickel alloy (50N).

The initial samples for the studies were contact plates 10x10 mm and 4 mm thick made of iron and nickel, wetted with tin and gallium, as well as round blanks with a diameter of 10 and 8 mm made of alloys 36N and 50N, respectively, with a central recess with a diameter of 4 by 5 mm for pouring liquid tin and gallium. After wetting (pouring) the samples with tin and gallium, they were subjected to isothermal holding for 10 to 50 h at a temperature of 300 and 500 °C. Then, sections were made for metallographic and other studies.

2. METALLOGRAPHIC STUDIES

After isothermal exposure, sections were prepared for metallographic studies on an Axio Observer microscope. The task of metallographic studies was to identify the number, shape and width of diffusion layers. To identify the microstructure of the base metal and new phases formed, etching was used with the following composition: H_2O_2 – 5 ml, NH_4OH – 25 ml, H_2O – 20 ml.

Previously, microsections were studied on a Carl Zeiss Jena optical microscope at magnifications of x200 and higher, and more detailed studies were conducted on a JEOL JSM 7001F scanning electron microscope (SEM) at an accelerating voltage of 10 and 15 kV, at different magnifications.

The distribution of chemical elements in the transition zone was studied using an energy-dispersive spectrometer. The detector of the energy-dispersive spectrometer is capable of registering up to 50 elements per measurement. The spectrometer allows the analysis of chemical elements from B to U.

Study of phase formation in the Fe-Sn and Ni-Sn systems

The microstructures of the transition zones obtained on a sample of iron and nickel after contact with liquid tin are presented in Fig. 1.

Study of phase formation in the Fe-Ga and Ni-Ga systems

The microstructures of intermetallic phases obtained on iron and nickel samples after contact with liquid gallium are presented in Fig. 2.

Study of phase formation in the (Fe-Ni)-Ga system

The microstructures of the diffusion layer obtained on samples of iron-nickel alloys 36H (Ni 36 % – Fe 64 %) and 50H (Ni 50 % – Fe 50 %) after contact with liquid gallium are presented in Fig. 3.

3. DETERMINATION OF MICROHARDNESS

Microhardness tests were carried out using the PMT-3 device. Measurements were made by indentation with a tetrahedral diamond pyramid under a load of 100 g. Each measurement was carried out at least three times. When calculating for each point, the arithmetic mean value was used. The microhardness was studied along the cross-section of the sample from the edge (source material) to the interaction zone (diffusion layer).

Fig. 4 shows diagrams of the distribution of microhardness values during the transition from the base metal to the intermetallic phase.

The following values were also obtained: changes in microhardness at the Fe-Ga phase interface at temperatures of 500 and 300 °C; changes in microhardness at the Ni-Ga phase interface at temperatures of 500 and 300 °C; changes in microhardness at the 50H-Ga phase interface at temperatures of 500 and 300 °C; changes in microhardness at the 36H-Ga phase interface at temperatures of 500 and 300 °C.

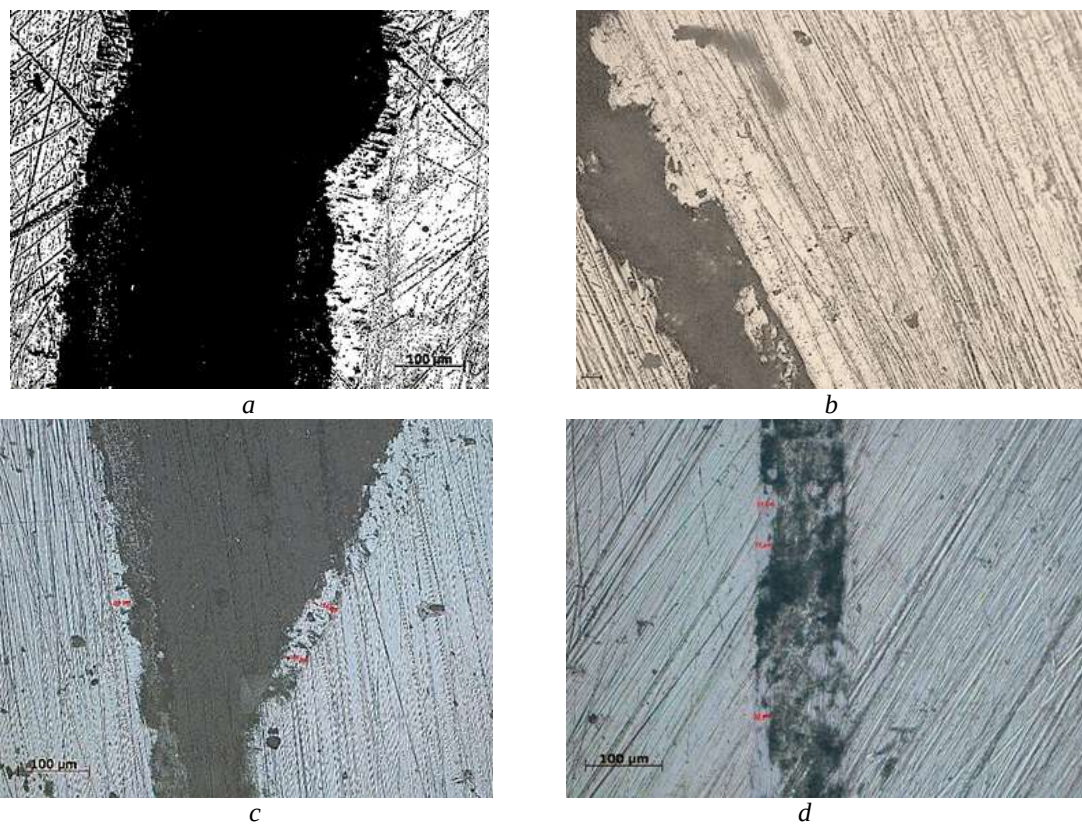


Fig. 1. Intermetallic layer obtained on samples:
a, b – on a sample of iron and nickel, respectively, after contact with tin at a temperature of 500 °C, holding time 50 h; c, d – on a sample of iron and nickel after contact with tin, at a temperature of 300 °C, holding time 50 h

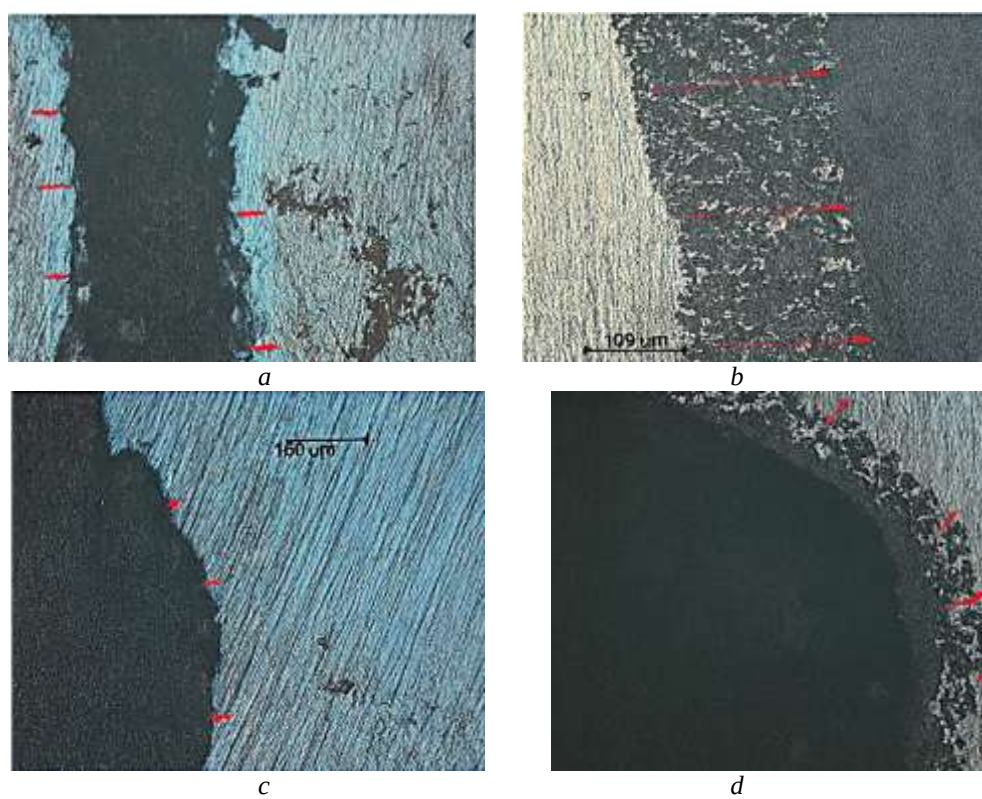


Fig. 2. Intermetallic layer obtained on samples:
a, b – iron and nickel, respectively, after contact with gallium, at $t = 500$ °C, holding time 50 h; c, d – iron and nickel, respectively, after contact with gallium, at $t = 300$ °C, holding time 50 h

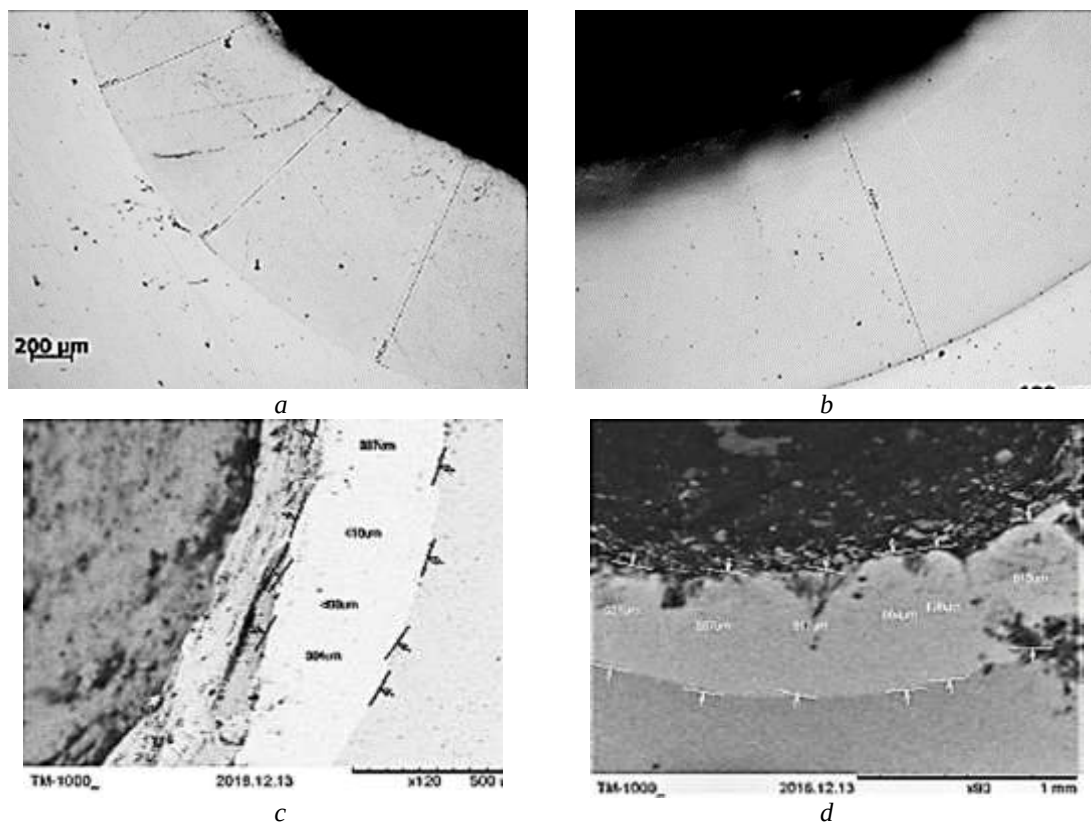


Fig. 3. Microstructure of the diffusion layer obtained on the samples:
a, b – alloy 36N and 50N, respectively, after wetting with liquid gallium, at $t = 500\text{ }^{\circ}\text{C}$, holding time 50 h;
c, d – 36N and alloy 50N, respectively, after wetting with liquid gallium, at $t = 300\text{ }^{\circ}\text{C}$, holding time 50 h

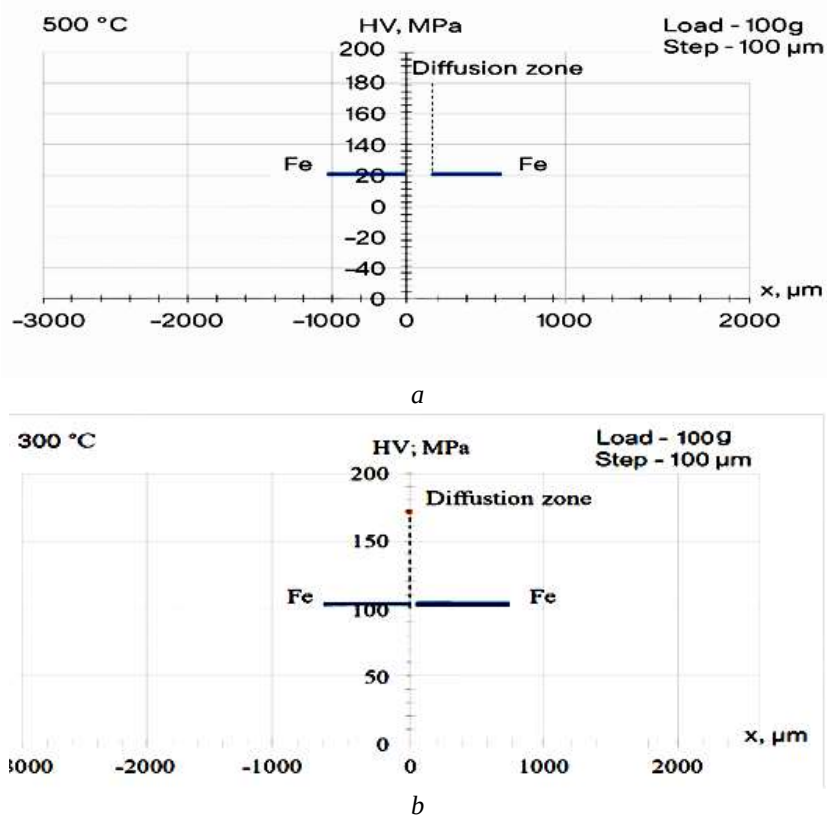


Fig. 4. Change in microhardness at the Fe-Sn phase interface:
a – at a temperature of $500\text{ }^{\circ}\text{C}$; *b* – at a temperature of $300\text{ }^{\circ}\text{C}$

4. STUDY OF THE ELEMENTAL AND PHASE COMPOSITION OF INTERACTION PRODUCTS BY THE METHOD OF ENERGY-DISPERSIVE MICROANALYSIS

The composition scanning step is 3 μm . According to the obtained data, concentration curves of the distribution of chemical elements along the scanning line were constructed in Excel.

When comparing the element concentration curves (Fig. 5) with the Fe-Ga diagrams, it can be stated that the FeGa_3 and Fe_3Ga_4 phases were formed. These compounds correspond to the element concentration on the diagram (Ga – 78.4%; Fe – 21.6%, and Ga – 61%, Fe – 39% (by mass)). Electron microscopic images of the transition zone of the 36H-Ga and 50H-Ga systems, respectively, were also obtained.

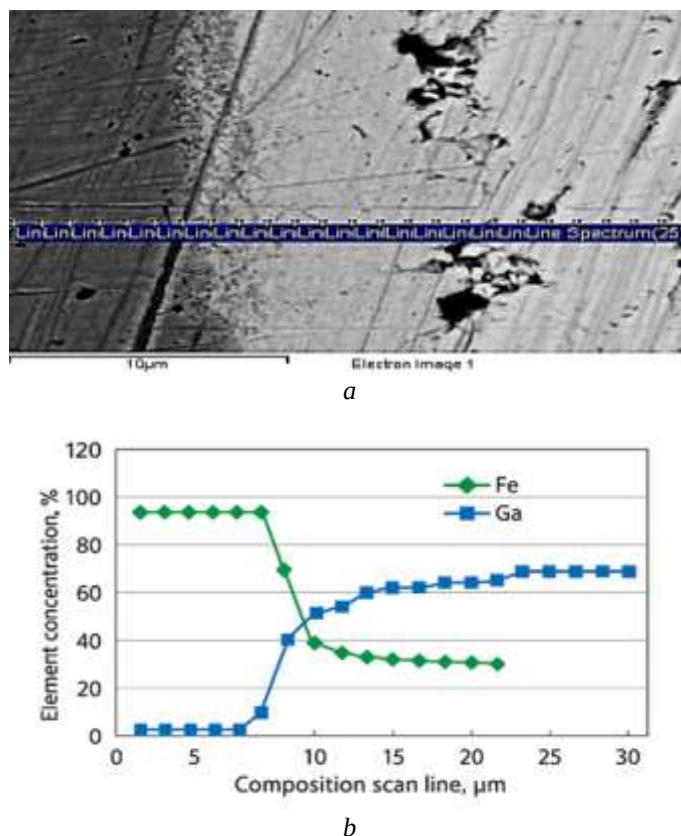


Fig. 5. Electron microscopic image of the transition zone in the Fe-Ga system (a) and concentration curves of the distribution of elements (b), $\times 5000$, linear spectra, step 3 μm

CONCLUSIONS

1. The kinetics of phase formation during the interaction of Fe, Ni and iron-nickel alloys 36H and 50H with liquid gallium and tin was studied. During the research, it was found that the formed intermetallics do not always grow according to a parabolic dependence in the coordinates: the thickness of the diffusion layer and the contact time.

2. It has been established that the rate of formation of new phases can be controlled when iron-nickel alloys interact with liquid gallium. Iron-nickel alloys with a high nickel content in gallium solder pastes can be used as solidification accelerators and as stabilizers (reduced Ni content).

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УДОСКОНАЛЕННЯ ПРИПОЮ ДЛЯ З'ЄДНАННЯ РІЗНИХ МАТЕРІАЛІВ

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Об'єктом дослідження були дифузійно-затверділі галієві припої та інші композитні компоненти. На ринку спостерігається активне зростання попиту на низькотемпературні технології складання, особливо в галузі Інтернету речей, носимих пристроїв та мікросенсорів. Зростаючий інтерес до низькотемпературних припоїв пояснюється тим, що вони мають дуже низьку температуру плавлення ($\approx 29,8^\circ\text{C}$), що робить їх перспективними для теплочутливих електронних компонентів, гнучкої електроніки, 3D-збірки без сильного нагрівання. Однак існують проблеми з їх використанням, які необхідно вирішити: корозія галію до багатьох металів, окислення поверхні, що призводить до поганої змочуваності, повільне затвердіння за кімнатної температури. Було проведено експериментальне дослідження фазоутворення під час взаємодії Fe, Ni та залізо-нікелевих сплавів 36Н і 50Н із рідким галієм і оловом. У ході дослідження було виявлено, що утворені інтерметаліки не завжди ростуть відповідно до параболічної залежності в координатах: товщина дифузійного шару та час контакту. Після розрахунку параметрів реактивної дифузії було встановлено, що ефективні коефіцієнти реактивної дифузії під час контакту галію в системах Fe-Ga, Ni-Ga вищі, ніж у системах Fe-Sn, Ni-Sn. Було встановлено, що під час взаємодії залізо-нікелевих сплавів із рідким галієм швидкість утворення нових фаз можна регулювати. Залізо-нікелеві сплави з високим вмістом нікелю в галієвих паяльних пастах можуть використовуватися як прискорювачі затвердіння, так і стабілізатори. Отримані результати дозволяють стверджувати, що функціональні припої є одним із напрямків сучасної мікроелектроніки.