PACS: 68.08.Bc, 68.08.De, 68.35.Np, 81.05.Je *DOI: 10.15407/Surface.2025.17.254*

WETTING AND CONTACT INTERACTION OF ZnO WITH Ag-Ge SYSTEM MELTS

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The aim of the work was to investigate wetting and contact interactions in pairs containing ZnO and Ag-Ge system melts on a macro scale. Sintered ZnO-ceramics without dopands of 15% porosity was used. Ag-Ge alloys were prepared by remelting. Wetting was studied by the sessile drop method. Wetting experiments were carried out at 1000°C in vacuum at germanium concentrations of 0, 1, 2, 3, 5, 10 and 100 at. %. The samples were photographed and the images obtained were processed using Adobe Photoshop. SEM investigations were carried out on the cross-sections of solidified on the ZnO surface metal drops using the Axia from Thermoscientific microscope with an energy-dispersive analyzer. Based on the data obtained, a graph of the dependence of wetting on the concentration of germanium was constructed. The addition of Ge to the Ag melt significantly improves its wetting of ZnO substrate (at 0 at. %. Ge θ=136°, at 2 at. % Ge θ=62, at 10 at. % Ge θ=41° and pure Ge spreads fully). There were dissolution of the substrate in the melt and diffusion of melt components into ZnO substrate. Both Ge and Ag diffusion in the depth of the substrate were observed. For drops of 1 and 2 at. % Ge, the concentration of Ge in ZnO substrate after the experiment was higher than in the drops. These phenomena are explained by the oxidation of Ge when reacting with ZnO. Oxygen in the composition of metal melts in contact with solid oxides is an adhesive-active component, as it forms complexes with metals that are adsorbed on interfaces, reducing intersurface tension, and thus improving wetting. In this case, oxygen-germanium complexes are formed and increase adhesion, then adsorbed Ge diffuses in ZnO. Diffusion of Ag is provided by formation of Zn vacancies in ZnO structure due to presence of Ge^{4+}. This effect can be used to increase the adhesion of metals to ZnO. At the same time, the interaction is very intense, this must be taken into account when developing devices containing tight contacts of the ZnO with metals. It is worth considering the possibility of using other elements that intensively interact with ZnO (Sn, In, Ga) as adhesive-active additives to inert metals (Ag, Au).

Keywords: *zinc oxide, metal melt, wetting, interface, contact interaction.*

INTRODUCTION

Zinc oxide (ZnO) is a wide-bandgap semiconductor with unique properties, which is prospective for designing catalysts, transparent conductive electrodes, transistors, etc. [1, 2]. In these applications, ZnO is often in close contact with a metal or metal-like semiconductor. Hence, the study of contact interaction in systems containing ZnO and metal is important, in particular for the development of metallization methods, as well as soldering and brazing. An effective method for such studies is wetting experiments, which allow evaluating adhesion and describing contact interaction in these systems. At the same time, the wetting of ZnO by metals on a macroscale has been studied in only a few works [3–7].

To provide high adhesion between a non-metal substrate and a metal melt, a chemical interaction is needed [8]. Ge nanoparticles [9] or thin films [10] react to ZnO at heating with Zn_2GeO_4 phase formation. At lower temperatures, GeO_x formed at the interface between ZnO film and germanium [11]. Ge can exhibit high adhesion to ZnO, in particular, acting as an addition to improving the wetting of the ZnO surface with metal melts. Ge addition was even used to improve the wetting of ZnO by Ag on a micro scale [12]. Therefore, the aim of the work was to investigate wetting and contact interactions in systems containing ZnO and Ge on a macro scale.

MATERIALS AND METHODS

Sintered ZnO-ceramics (further as “ZnO substrate”) without dopands with a porosity of 15%, high-purity Ag (0.999) and Ge (0.999) were used. The surface of the ZnO substrate was polished with 0.7-0.3 μm diamond paste. Ag-Ge alloys were prepared by remelting in vacuum in alumina crucibles.

Wetting was studied by the sessile drop method [8]. The experiments were performed in a vacuum; the 40 L chamber was equipped with a water-cooling system and a window to observe the samples. A vacuum of not lower than 3×10^{-3} Pa was provided by diffusion and backing pumps. The heating of the experimental samples was carried out in a resistance furnace with heaters in the form of tungsten 0.1 mm plates and molybdenum screens.

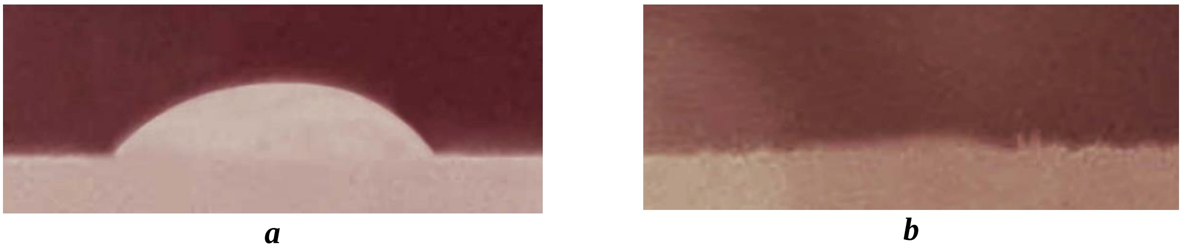
The samples were photographed through the window in the wall of the vacuum chamber; the images were processed using Adobe Photoshop.

SEM investigations were carried out on the Axia from Thermoscientific microscope with an energy-dispersive analyzer.

RESULTS AND DISCUSSION

First, the wetting of ZnO with pure germanium was investigated (Fig. 1). The melt wetted the ZnO (Fig. 1*a*) and completely spread over its surface within 5 min (Fig. 1*b*).

In this case, germanium completely reacted with zinc oxide, the formed loose reaction products are difficult to analyze, so Ag-Ge alloys with low concentrations of germanium were further investigated. Ag was chosen as the base metal because, firstly, it has a relatively low melting point, secondly, it interacts very weakly with ZnO [13–15], unlike, for example, Cu [16–21] or Sn [6, 7] and thirdly, because on a micro scale, the wetting of the ZnO with silver was improved by Ge adding [12].



a ***b***

Fig. 1. Wetting of ZnO-ceramics with Ge melt with various holding times at 1000°C: *a* – 0; *b* – 5 min

Wetting experiments were performed at 1000°C in vacuum, germanium concentrations of 0, 1, 2, 3, 5 and 10 at. % were considered. Equilibrium contact angles were reached within 10-12 min, a 15 min holding was used to confirm this. The resulting photographs of the drops are presented in Fig. 2; a graph of wetting on the concentration of germanium dependence is presented in Fig. 3.

Ge has a very significant effect on wetting (Fig. 2, and Fig. 3), the addition of 1 at. % Ge to Ag reduces the contact angle from 136 (Fig. 2*a*) to 98° (Fig. 2*b*), and 2 at. %, respectively, to 60°, i.e., there is a transition from non-wetting to wetting. Further increase in the germanium content improves wetting not so significantly, but noticeably (Fig. 3, Fig. 2*d*, Fig. 2*e*); at 10 at. % Ge, a contact angle of 40° was reached (Fig. 2*f*). Therefore, in the system studied, Ge is an adhesion-active component. The shape of the Ag-1Ge (Fig. 2*c*) and Ag-3Ge (Fig. 2*e*) drops deviates from the regular spherical one. This is typical for the wetting of polycrystalline porous ceramics by metal melts with a low concentration of the active component [8] but may also indicate a heterogeneous composition of the melt due to intensive interaction with the substrate.

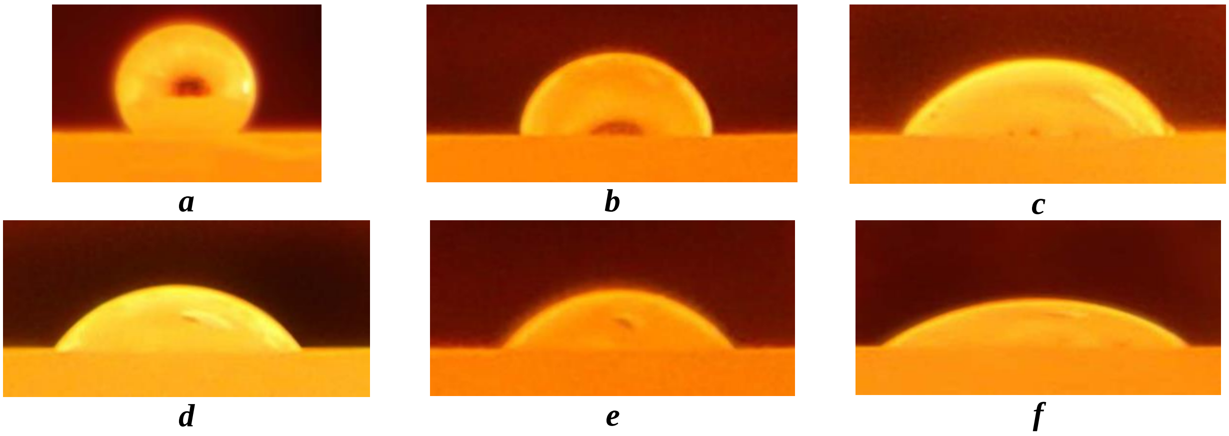


Fig. 2. Ag-Ge drops with different Ge concentration on the surface of ZnO-ceramics after reaching of equilibrium contact angle, 1000°C, vacuum, 15 min holding: *a* – pure silver; *b* – Ag-1Ge melt; *c* – Ag-2Ge melt; *d* – Ag-3Ge melt; *e* – Ag-5Ge melt; *f* – Ag-10Ge melt

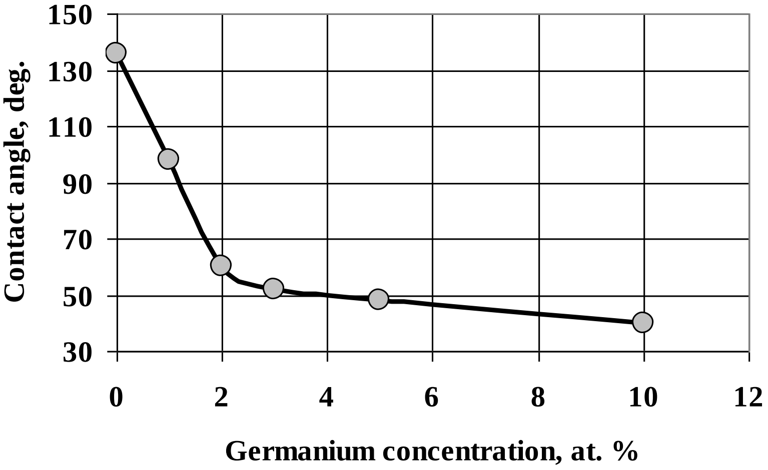


Fig. 3. Wetting of ZnO-ceramics by Ag-Ge melts, 1000°C, vacuum, 15 min holding

The drops of the Ag-5Ge and Ag-10Ge compositions broke off from the substrates due to the formation of loose transition layers at the interface. The samples with Ag-1Ge and Ag-2Ge drops were also destroyed, but the cracks passed through the ceramics; the sample with the Ag-3Ge drop remained intact, so cross-sections were made and the interfaces examined by SEM. Results for the Ag-1Ge drop case are represented in Fig. 4 for an interface at the middle of the drop base and in Fig. 5 for the triple line area.

The interface is very uneven (Fig. 4*a*, Fig. 5*a*), there are signs of infiltration of the melt into the substrate (Fig. 5*a*). In addition, Zn is present in a small amount in the solidified drop (Fig. 4*b*, Fig. 5*b*), this is especially noticeable in Fig. 5*b*, when compared the zone of the metal drop with the upper left corner of the image, corresponding to epoxy resin, which was used to pour over the samples at the fabrication of the cross-sections. The oxygen of the substrate was also apparently dissolved in the melt (Fig. 4*c*, Fig. 5*c*), its contents in the drop and in the epoxy resin are similar (Fig. 5*c*). So, ZnO dissolved in the melt. That is, the drops “deepened” into the substrate, and the visible contact angles measured from the photographs of the drops do not correspond to the real ones; the curve in Fig. 3 should pass higher.

Germanium is present in both the solidified drop and the substrate (Fig. 4*d*, Fig. 5*d*). Moreover, its content in the substrate is higher than in the drop, that is, a kind of "extraction" has occurred.

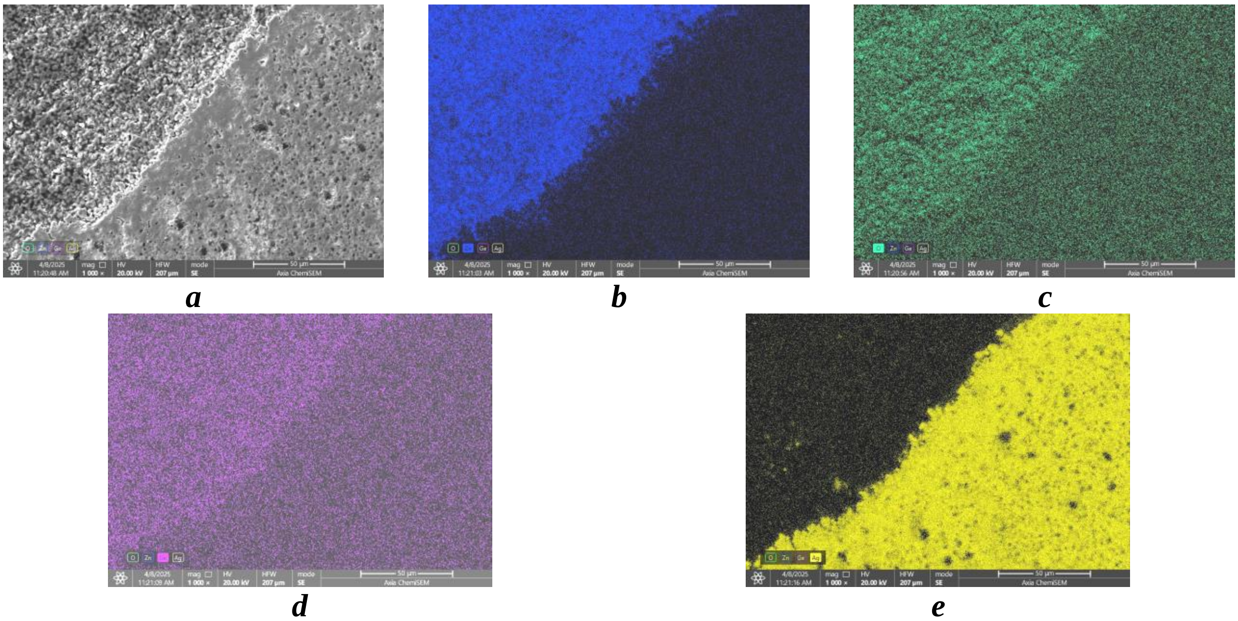

a ***b*** ***c***
d ***e***

Fig. 4. Microstructure and elements distribution map of the interface area for Ag-1Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

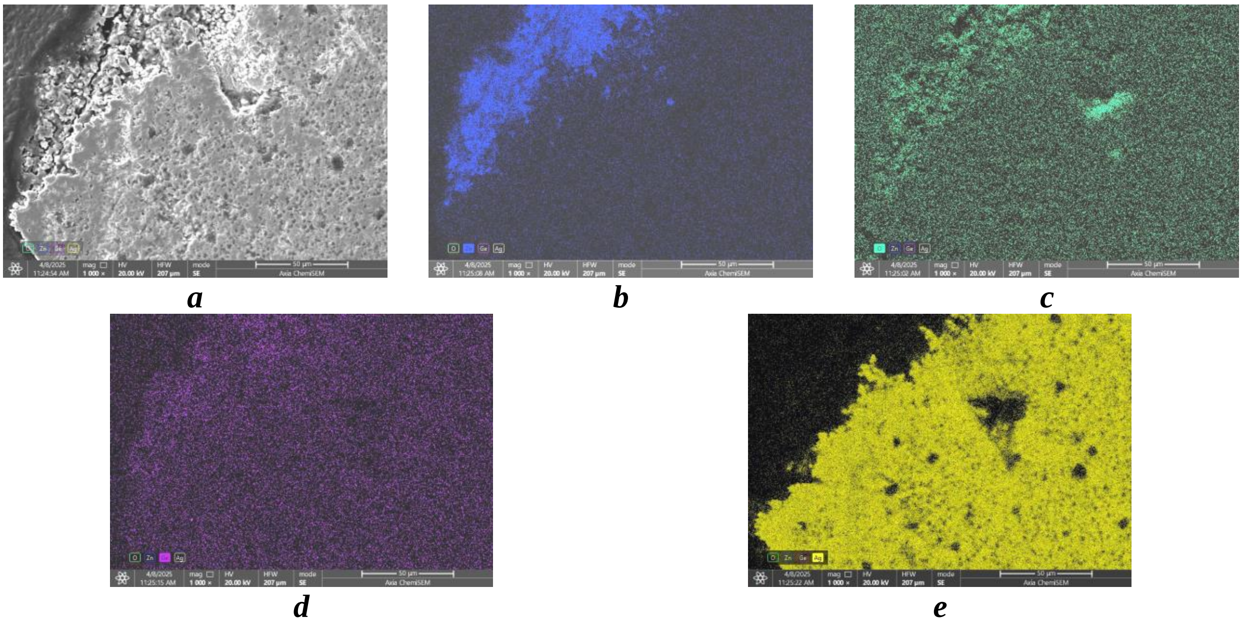

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Fig. 5. Microstructure and elements distribution map of the triple line area for Ag-1Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

Results of the SEM study of the sample with Ag-2Ge drop are represented in Fig. 7 for an interface at the middle of the drop base and in Fig. 6 for the triple line area.

The surface of the ZnO substrate was also destroyed by interaction with the metal melt, zinc (Fig. 6*b*, Fig. 7*b*) and oxygen (Fig. 6*c*, Fig. 7*c*) were dissolved in the drop from the substrate, and germanium was "extracted" from the melt deep into the zinc oxide (Fig. 7*d*). In this sample, silver is also present in the substrate layer near the interface (Fig. 6*e*, and Fig. 7*e*). The far from interface boundary of the silver containing ZnO layer is clear, i.e. the silver was not brought in during polishing, but penetrated into the substrate probably by diffusion, which is theoretically possible [14, 15, 22].

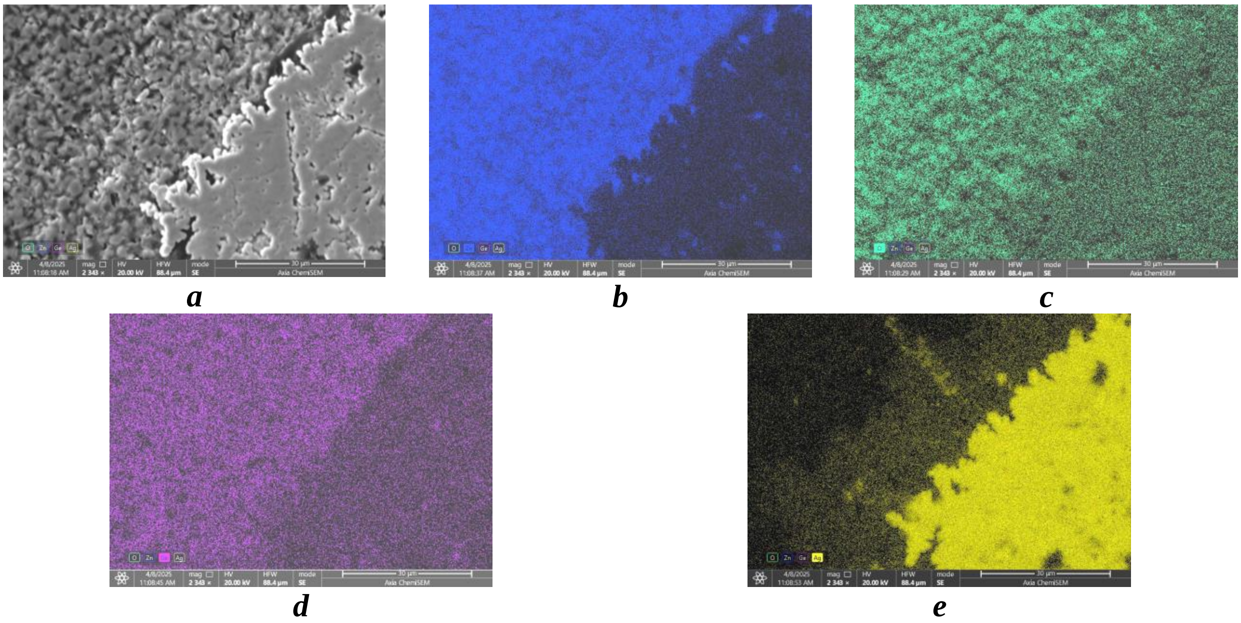

Fig. 6. Microstructure and elements distribution map of the interface area for Ag-2Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

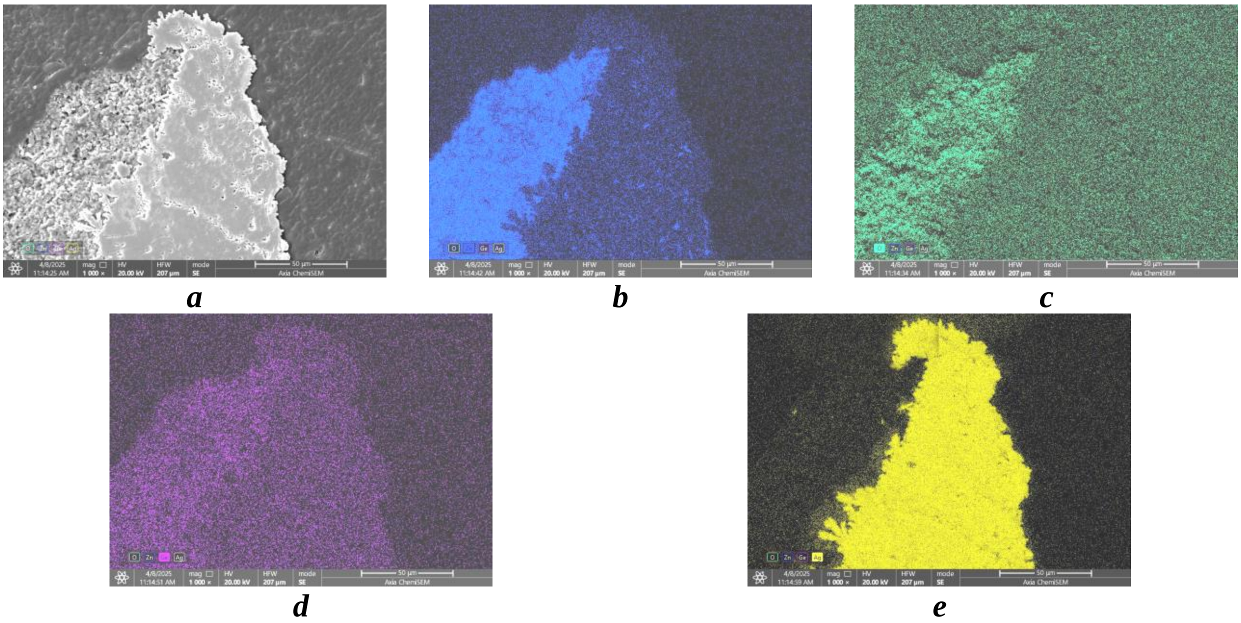

Fig. 7. Microstructure and elements distribution map of the triple line area for Ag-2Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

SEM investigation’s results for the sample with the drop of Ag-3Ge composition are represented in Fig. 8 for the middle of the drop base and in Fig. 9 for the triple line area.

The ZnO substrate is also destroyed due to interaction with the melt (Fig. 8*a*, and Fig. 9*a*), zinc (Fig. 8*b*, and Fig. 9*b*), and oxygen (Fig. 8*c*, and Fig. 9*c*), which are also dissolved in the drop. As in the previous samples, Ge penetrated ZnO substrate, but this time its content in the solidified drop is higher than in the substrate (Fig. 8*d*, Fig. 9*d*), probably, the maximum concentration of Ge in zinc oxide was reached, according to the data of the work [23] the solubility of Ge in ZnO is 0.7 mol %. Ag (Fig. 8*e*, Fig. 9) penetrated into ZnO substrate much deeper than in the case of the sample with a drop of the Ag-2Ge composition (Fig. 6*d*, Fig. 7*d*).

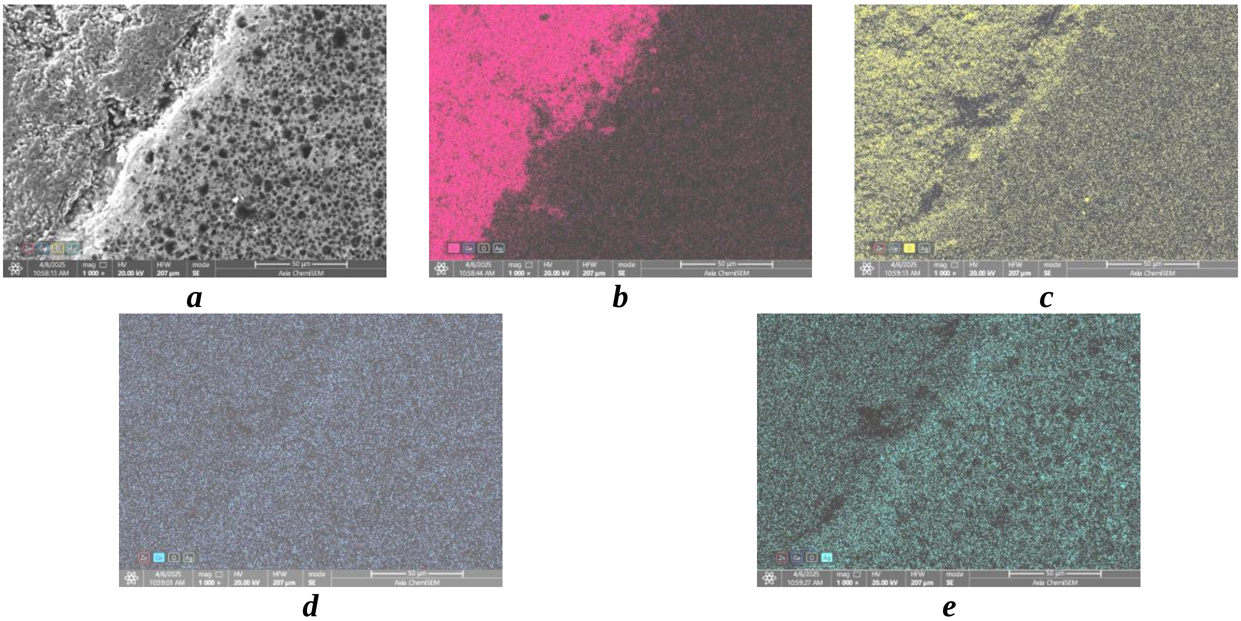
a ***b*** ***c***

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Fig. 8. Microstructure and elements distribution map of the interface area for Ag-3Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

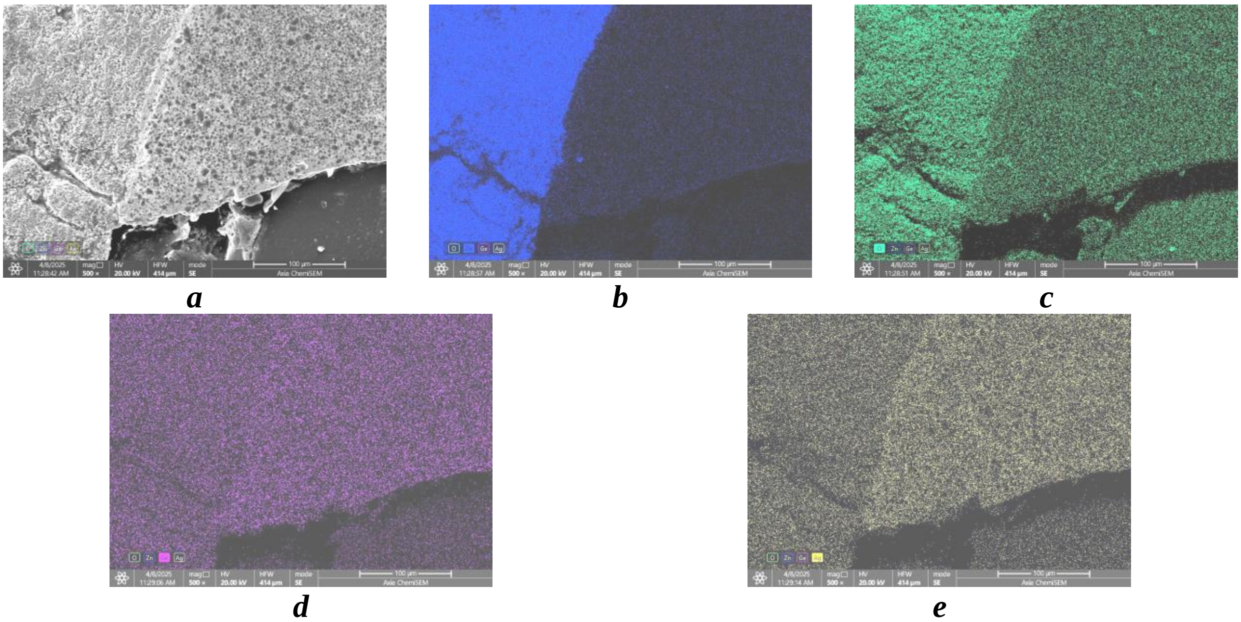
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Fig. 9. Microstructure and elements distribution map of the triple line area for Ag-3Ge drop solidified on the ZnO-ceramics surface: *a* – microstructure; *b* – Zn distribution; *c* – O distribution; *d* – Ge distribution; *e* – Ag distribution

The structure of the solidified drops is complex; it is a mixture of phases with different contents of the components of the system under study (Zn, O, Ag, and Ge). This is especially noticeable for the Ag-3Ge drop (Fig. 8, Fig. 9), whose structure is an Ag-rich matrix and Ag-depleted inclusions. Given that the solubility of Ge in solid silver decreases with cooling [24], it can be assumed that the drop solidified as a solid solution of Ge in Ag, from which, with a further decrease in temperature, Ge was released, forming the mentioned inclusions. The presence of Zn also probably reduces the solubility of Ge, since there are no compounds or regions of solid solutions in the Zn-Ge system [25].

Since pure Ag does not wet ZnO, and the addition of Ge leads to intensive dissolution of the ZnO substrate in the melt and a significant improvement in wetting, in addition, Ge penetrates into ZnO, it is obvious that it is Ge that interacts with ZnO.

As already mentioned, there are no compounds of Ge with Zn, therefore, germanium oxidation occurred, as in [9-11]. Using the resource [26], the Gibbs energies of reactions $2ZnO + Ge = 2Zn + GeO_2$ and $ZnO + Ge = Zn + GeO$ were calculated; they are equal to 84.27 kJ/mol and 55.69 kJ/mol, respectively, that is, the probability of these processes is small. However, it is worth noting that reactions could also occur on the interface, where Zn dissolves in drops, and oxidized germanium diffuses into the ZnO substrate, i.e., the reaction products leave the reaction zone, which shifts the balance towards their formation.

The observed effects can also be explained by the tendency of ZnO to form non-stoichiometric phases with oxygen deficiency during annealing in vacuum [27, 28], i.e., the interaction with metal melts is facilitated by the presence of a significant number of defects in the structure of ZnO [16, 21]. Contact with the active component (germanium) in the composition of the metal melt increases the probability that, as a result of interaction with it, oxygen will leave the oxide structure and dissolve in the melt. This process may be described by the equation $ZnO + Ge = ZnO_{1-x} + GeO_x$.

Oxygen in the composition of metal melts in contact with solid oxides is an adhesive-active component, as it forms complexes with metals that are adsorbed on interfaces, reducing intersurface tension, and thus improving wetting [8]. In this case, oxygen-germanium complexes are formed and increase adhesion.

Germanium is probably oxidized to Ge^{4+}, since this was observed in [9, 10], so its diffusion deep into the substrate leads to the formation of cationic vacancies in the structure of zinc oxide, which, in turn, contributes to the diffusion of silver into ceramics [29], which signs have been observed in microstructure studies.

Thus, the interaction of ZnO with the Ag-Ge melt is a complex process with the dissolution of the oxide components in the metal and the diffusion of the melt components into the oxide.

CONCLUSION

The addition of Ge to the Ag melt significantly improves the wetting of ZnO due to the oxidation of Ge when interacting with ZnO. This effect can be used to increase the adhesion of metals to ZnO.

At the same time, the interaction is very intense, there is dissolution of ZnO substrate in the melt, and the penetration of the melt components deep into the substrate. This must be taken into account when developing devices containing tight contacts of the ZnO with metals.

It is worth considering the possibility of using other elements that intensively interact with ZnO (Sn, In, Ga) as adhesive-active additives to inert metals (Ag, Au).

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PACS: 68.08.Bc, 68.08.De, 68.35.Np, 81.05.Je DOI: 10.15407/Surface.2025.17.254

ЗМОЧУВАННЯ ТА КОНТАКТНА ВЗАЄМОДІЯ ZnO З РОЗПЛАВАМИ СИСТЕМИ Ag-Ge

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Метою роботи було дослідження змочування та контактної взаємодії у парах, що містять ZnO та розплави системи Ag-Ge, на макрорівні. Використовували спечену ZnO-кераміку без домішок пористістю 15%. Сплави Ag-Ge готували переплавленням. Змочування вивчали методом лежачої краплі. Експерименти зі змочування проводили при 1000°C у вакуумі при концентраціях германію 0, 1, 2, 3, 5, 10 та 100 ат. %. Зразки фотографували, а отримані зображення обробляли за допомогою Adobe Photoshop. SEM-дослідження проводили на шліфах затверділих на поверхні ZnO крапель металу за допомогою мікроскопа Thermoscientific Axia з енергодисперсійним аналізатором. На основі отриманих даних побудовано графік залежності змочування від концентрації германію. Додавання Ge до розплаву Ag значно покращує змочування ним підкладки ZnO (при 0 at. %. Ge θ=136, при 2 at. % Ge θ=62, при 10 at. % Ge θ=41° та чистий Ge розтікається повністю). Відбувалося розчинення підкладки в розплаві та дифузія компонентів розплаву в підкладку ZnO. Спостерігалася дифузія як Ge, так і Ag в глибину підкладки. Для крапель з 1 та 2 ат.% Ge концентрація Ge в підкладці ZnO після експерименту була вищою, ніж у краплях. Ці явища пояснюються окисленням Ge при реакції з ZnO. Кисень у складі металевих розплавів, що контактують з твердими оксидами, є адгезійно-активним компонентом, оскільки він утворює комплекси з металами, які адсорбуються на межі розділу, зменшуючи міжповерхневий натяг і тим самим покращуючи змочування. При цьому утворюються киснево-германієві комплекси, які збільшують адгезію, потім адсорбований Ge дифундує в ZnO. Дифузія Ag забезпечується утворенням вакансій Zn у структурі ZnO завдяки наявності Ge^{4+}. Цей ефект може бути використаний для збільшення адгезії металів до ZnO. Водночас взаємодія дуже інтенсивна, це необхідно враховувати при розробці пристроїв, що містять щільні контакти ZnO з металами. Варто розглянути можливість використання інших елементів, які інтенсивно взаємодіють з ZnO (Sn, In, Ga), як адгезійно-активних добавок до інертних металів (Ag, Au).

Ключові слова: *оксид цинку, металевий розплав, змочування, поверхня розділу, контактна взаємодія.*