Wettability by Liquid Metals, Metalization, and Brazing of Barium Titanate Ceramics

A study investigates the contact interaction and wetting of BaTiO3 perovskite ceramics by liquid metals

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ABSTRACT

Detailed investigations, including wetting studies by liquid metals and metal coatings deposition onto ceramic surfaces and brazing processes, were carried out for semiconducting and ferroelectric perovskite ceramic states of barium titanate (BaTiO3). Pure metals (Cu, Ag, Al, Ge, Sn, Pb, Ga, In, Al, Si, Ni, Co, Fe, Pd) and Ti-containing alloys based on In and binary Cu-Sn, Cu-Ga, and Cu-Ag systems were investigated under high vacuum for the semiconducting BaTiO3-x surface. The degree of wettability correlates approximately with the chemical affinity of the liquid metal phase to oxygen (wetting increases when the liquid metal affinity to oxygen increases). Addition of Ti to the liquid metal phase increases capillary properties and adhesion in the system under investigation. Investigations of the wettability of ferroelectric BaTiO3 ceramics were performed in air gaseous media by alloys Ag-Cu-O. Oxygen in the gaseous media preserves stoichiometric composition of barium titanate and being dissolved in Ag-Cu alloys promotes BaTiO3 wetting. For the first time, wettability experiments in liquid metal/ceramic material systems (BaTiO3 in this case) were carried out when the gaseous phase was pure oxygen. At greater oxygen partial pressure (1 atm for O2 comparing to 1/5 atm for air), wettability further increases significantly. Vacuum brazing technology for semiconducting materials and joining processes in air or pure oxygen atmosphere for ferroelectric ceramic materials based on BaTiO3 have been developed. Various detailed brazing models of BaTiO3 and BaTiO3-x were created.

Introduction

The perovskite-type structure ceramic materials, such as barium titanate, play a major role in modern electronics and electrical engineering. Barium titanate (BTO) is widely used for creating multilayer ceramic capacitors (MLCCs), embedded decoupling capacitors (EDC), electrical ceramic filters, and other piezoelectric and ferroelectric components (Refs. 1–3). Ferroelectric ceramics are ideal for use in supercapacitors. On the base of these materials, a ferroelectric memory device was created (Refs. 4, 5). Barium titanate is also an excellent photorefractive material (Ref. 6).

Oxygen release that occurs during the annealing of BaTiO3 in high vacuum at sufficiently high temperatures leads to the transformation of dielectric ferroelectric BaTiO3 into semiconducting ceramics (Ref. 7). As a semiconductor, BaTiO3 particularly exhibits a positive temperature coefficient of resistivity (PTCR). It means that at a certain temperature (Curie temperature), this material exhibits strong resistivity increase (typically by several orders of magnitude) (Ref. 6); due to its PTCR properties, barium titanate is often used as thermistors material in the thermal switches. The great importance for all such ceramics is creation of strong contacts (including electric ones) in combinations BaTiO3/metals and BaTiO3/BaTiO3.

Strength of metal-oxide contact and uniformity of metal coating are determined essentially by a wettability degree of ceramic materials (BTO) surface by liquid metals. High adhesion of liquid metals to ceramics surface is a crucial factor for creating mechanically strong contact.

According to Ref. 8, the degree of perovskite-type ceramics wettability by liquid metal, and intensity of interaction between liquid metal and solid phase, can also determine some electric properties of the contact, e.g., ohmic or nonohmic one, p-n-transition (in the case of semiconducting ceramics), and Schottky barrier height.

Thus, creating a strong adherent metal coating on the BaTiO3 surface for its joining to metals and metalization of perovskite-type ceramics is a perspective direction of investigation. Recently, scientific and technical interest in this problem was increased considerably.

Up to now, there are only a few published works concerning research of contact properties of some metals in relation to perovskite-type ceramics, in particular to barium titanate (Refs. 9–11). The experimental data on wetting of perovskite ceramics by some pure metals in these works contradict each other sometimes. In addition, in Ref. 10 the conditions of experiments are described only qualitatively, e.g., as atmosphere with “high” and “low” oxygen pressure, so these data need verification. Regularities of strong adherent contact formations are studied little; scientific bases of these processes are practically absent.

The present work aims to systematically investigate the details of phenomena for wettability, adhesion, and interaction intensity of BaTiO3 perovskite-type ceramics in different forms — ferroelectric...
and semiconducting — with molten metals that will allow doing further steps in understanding metal-perovskite ceramics interaction and elaboration of some brazing alloys and technological processes for joining (brazing) of BaTiO₃ materials.

**Semiconducting BaTiO₃₋ₓ**

Nonstoichiometric semiconducting BaTiO₃₋ₓ can be obtained by means of annealing in high vacuum, as is mentioned above. It is believed that BaTiO₃₋ₓ nonstoichiometry is insignificant and will not exceed such parameters for pure titanium oxide TiO₂₋ₓ. For this oxide, the x value is between 0.04 and 0.07 (Ref. 12). Such deviation only has a minor effect on mechanical and thermodynamic properties of the compound, except for the electrophysical characteristics.

Such nonstoichiometry variation can be attributed to oxygen vacancies compensated mostly by background and/or intrinsic acceptors within higher oxygen partial pressure (p(O₂)) regions and by electrons within lower p(O₂) regions (Ref. 13). Semiconducting BTO has specific resistivity value near 300 Ω·cm (compared to about 10⁸–10¹⁰ Ω·cm for ferroelectric BaTiO₃).

The technology of vacuum metalization and brazing by melts containing titanium as a chemically active element was tested for preliminary annealed semiconducting barium titanate ceramics.

**Experimental and Discussion**

The main experiments consist of the wettability measurements of BTO by liquid metals. Wettability studies were carried out by a sessile drop method in vacuum (~ 10⁻⁴ Pa) at temperature 870–1870 K. This method allows determining the values of the wetting contact angle and interfacial surface energy at the liquid-gas interface. The sessile drop method essence was discussed in detail earlier (Refs. 14–16). The main requirement for measuring wetting contact angle by the sessile drop method is in the placement of a symmetrical drop of the melt on the solid surface. The sample should be in controlled gaseous atmosphere or in at the temperature specified. Standard equipment for the wettability of solid ceramic specimens by a liquid metals study using the sessile drop method is shown in Fig. 1.

A wide variety of metals and alloys having a broad application range in electroceramic devices was used. Fourteen pure metals (Cu, Ag, Au, Ge, Sn, Pb, Ga, In, Al, Si, Ni, Co, Fe, Pd) and several titanium-containing alloys (Cu-Sn-Ti, Ag-Cu-Ti, Cu-Ga-Ti, In-Ti) were tested.

Metal samples for wetting experiments typically have approximately 0.5–0.9 g. Metal alloys were formed in-situ by alloying. Polycrystalline barium titanate has been specially fabricated by the method of solid-phase synthesis. In this study, we used BaTiO₃ ceramic discs 20 mm in diameter and ~ 3 mm thick. The sample’s porosity was 3.5±0.03%. BTO substrates were ground and polished with sandpaper and abrasive powder. The average surface roughness value (Rₐ) was equal to 0.02 μm. Before experiments, BTO samples were annealed in vacuum at ~ 1740 K during 60 min.

The wetting of BTO by molten alloys Cu 8.6% (at.) Sn, Ag 39.9% (at.) Cu, and Cu 17.6% (at.) Ga (which was used to create the many braze alloys) with active titanium additive (from 3 up to 25% (at.)) was studied as well. Results of the wetting studies of BTO by pure metals melts and some alloys are presented in Table 1.

Most of the investigated pure metals did not wet the barium titanate ceramics surface (contact angles exceeded 90 deg). Silicon and aluminum wet BTO (alu-
minum has a minimum value contact angle of 78 deg). As a whole, the results agree with Ref. 9. For example, wetting contact angle values for Ag and Au in Ref. 9 confirm our data, though we consider value of contact angle for Cu as obviously underestimated.

Overall, a high degree of solids wetting by liquid metals is caused by an intensive interfacial chemical interaction (Figs. 2, 3). For oxides like Al₂O₃, SiO₂, and MgO, liquid metal interaction with oxygen is the main factor (Refs. 17, 18). Comparatively, due to the more anion O²⁻ dimension to metal cation one and the more latter displacement in the bulk of crystal (effect of relaxation of surface ions and according to Weyl’s scheme for structure of surface of oxides), the oxide surface is formed mainly by oxygen anions.

The BTO surface structure containing two cations of different sizes and valence numbers required special consideration. Titanium ions (with a 0.068-nm radius) are

Fig. 4 — Microstructure of (Cu-8.6 Sn) – 25 Ti-BaTiO₃ interface. A — ×1000; B — distribution of elements at interface, % (at.); and C — characteristic emission of elements at BaTiO₃ molten metal interface (top part – ceramics, bottom part – alloy) with barium (1), titanium (2), tin (3), and copper (4).
located in octahedral cavities formed by oxygen ions and have enough “room” for displacement within the BTO elementary cell (a = 4.011 for cubic lattice). It is an explanation of essential mobility for titanium ions oscillating freely within the octahedral environment of oxygen ions. It determines high polarizability of barium titanate under electric field action (Ref. 19). Such increased mobility of titanium ions (according to Weyl’s scheme (Ref. 20)) leads to some more of their displacement within the BTO elementary cell (a = 0.135 nm, r(O2–) = 0.140 nm) and structure of BTO crystal lattice, we can see that only a quarter of the ceramic’s surface is occupied by Ba2+ ions, and the rest by oxygen ions. Overall, we can guess that main regularities of interactions in BaTiO3 liquid metal systems have to be similar to regularity for “classical” oxide (Al2O3) metal systems. Nevertheless, the interaction of certain liquid metal phases with barium ions at a BaTiO3 surface should be considered.

Free formation energies of chemical compounds for the metals under investigation with barium are within 167–250 kJ/mol (for comparison, heat of formation for the oxides is ΔH(Al2O3) = –1675 kJ/mol, ΔH(SiO2) = –911 kJ/mol) (Ref. 21). Only a silicon compound with barium (BaSi3) is formed with significant heat release ΔH(BaSi3) = –544 kJ/mol). But pure silicon can only moderately wet the BaTiO3 surface, and its adhesion is lower than the same value for aluminum, though Al-Ba compounds are considerably less stable thermodynamically according to phase diagrams data (Ref. 22). Thus, wetting and adhesion in BTO metal systems is evidently not determined by Ba-Me interaction to a significant degree.

An inactive matrix of Cu-Ga, Ag-Cu, and Cu-Sn alloys (Fig. 3) does not wet the BTO surface (θ = 120–130 deg). A titanium addition reduces contact angles down to 20–70 deg for titanium concentration up to 10–25% (at.). It has been assumed that wetting the BTO surface is, first of all, a result of interaction between liquid metal (titanium) and oxygen of solid phase, as in the case of “classical” oxide materials (Al2O3 and MgO). Formation of titanium oxide (TiO) having metal-like properties in a BaTiO3/Ti-containing alloy system is the reason of high adhesion in this case. Titanium, as a transition metal, is characterized by its ability to participate simultaneously in several chemical bonding interactions of different types — ionic one with BaTiO3 surface and metallic with liquid metal phase. In other words, titanium from a liquid phase can become a bridge connecting the solid BaTiO3 phase with molten metal.

Our SEM research of contact boundary BaTiO3/titanium-containing alloy has shown the presence of a transitive zone 5–7 μm wide, which is obviously a product of interphase reaction. Figure 4 shows the structure of cooled drop (Cu-8.6 Sn)-20Ti on the BaTiO3 substrate.

Analysis of the BaTiO3/liquid metal interface shows the character of elements distribution in the direction perpendicular to the interface — Fig. 4B. Chemical composition for the BaTiO3 phase in volume is reproduced precisely as ~20% (at.) of barium and titanium and ~60% (at.) of oxygen. This ratio remains unchanged to the BaTiO3/metal interface. Barium concentration is insignificant at the interface. That is why metal interaction with barium is possible only as a monolayer adsorption at the BaTiO3 surface. Titanium concentration increases from 20% (at.) in the transition zone up to ~50–60% (at.) in the contact zone. Oxygen concentration in this zone is about 18% (at.).

In Fig. 4C, the layer (new phase) with high titanium concentration is clearly visible. Titanium segregation from the melt at interface is the main reason for high wettability of BaTiO3 by Ti-containing alloys. The metal chemistry studies by M. V. Nevitt (Ref. 23) show that oxygen stabilizes intermetallic compounds like Ti3Cu; the Cu23Ti33O phase has been identified. A special investigation of the processes occurred at different temperatures in the contact zone by a high-temperature, X-ray diffraction method of pressed mixture with barium titanate, copper, and titanium powders carried out as well (Fig. 5).

Two new phases with TiO and Cu3Ti3O structures were identified in this system. Both substances can be responsible for wetting. However, the Cu3Ti3O pattern disappears at 1370 K (Fig. 5B); probably, this compound is not stable. Just TiO can

<table>
<thead>
<tr>
<th>Metal</th>
<th>Temperature, K</th>
<th>Contact Angle θ, deg</th>
<th>Work of Adhesion, MJ/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>1373</td>
<td>122±3</td>
<td>610</td>
</tr>
<tr>
<td>Ag</td>
<td>1253</td>
<td>136±3</td>
<td>260</td>
</tr>
<tr>
<td>Au</td>
<td>1273</td>
<td>132±1</td>
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</tr>
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<td>Ge</td>
<td>1273</td>
<td>129±2</td>
<td>345</td>
</tr>
<tr>
<td>Sn</td>
<td>873</td>
<td>120±1</td>
<td>285</td>
</tr>
<tr>
<td>In</td>
<td>673</td>
<td>152±1</td>
<td>70</td>
</tr>
<tr>
<td>Pb</td>
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<td>117±2</td>
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<tr>
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<td>1423</td>
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<td>1015</td>
</tr>
<tr>
<td>Co</td>
<td>1793</td>
<td>78±2</td>
<td>1140</td>
</tr>
</tbody>
</table>

Table 1 — The Results of Wetting of Semiconducting Barium Titanate by Some Pure Metals

* WELDING RESEARCH
be accounted for the explanation of a wetting-in system studied at high temperatures. The same data were published, for example, in Ref. 24 for Al2O3 systems.

Varying wetting effects of titanium in different systems studied — Cu-Ag, Cu-Ga, Cu-Sn (Fig. 3) — can be explained by several reasons, in particular by various thermodynamic activity of titanium in alloys. Ag-Cu-Ti alloys demonstrate some peculiarity. A eutectic system with about 60% (at.) of silver can dissolve in about 2% (at.) of titanium at 1270 K. A titanium content increase leads to arising the second equilibrium liquid phase that contains 64% (at.) of Cu, 28% (at.) of Ti, and 8% (at.) of Ag (Ref. 14). The mechanism details of such interface processes in a complex BaTiO3 – (Ag–Cu–Ti) phase I – (Ag–Cu–Ti) phase II system requires special consideration. It is possible now to note only that this process can be useful for improving adhesive bonding of BaTiO3 to metal (arising of second liquid phase with a high Ti concentration).

The temperature dependence of contact angle for indium-titanium alloys on the BaTiO3 surface has been investigated as well. The contact angle of an In-Ti melt drops significantly at a low temperature (in interval, ~770–870 K). Almost full spreading of the In-Ti melt on a BTO surface occurs at 830–870 K. It can be used for brazing not only semiconducting barium titanate (BaTiO3) but the ferroelectric one (BaTiO3) as well.

Brazing Alloys and Technological Conditions for Semiconducting BTO Joining

Metalization of materials using liquid metal film is a perspective method. However, a high degree of wetting for solid surfaces by such metal is required. Theoretically (Ref. 14), for producing continuous film of liquid metal, spreading factor (K) has to be positive (Equation 1).

\[ K = W_A - W_C \]  

where \( W_A \) is work of adhesion and \( W_C \) is work of cohesion.

For brazing and metalization of perovskite, compounds were chosen for brazed compositions that are well wetted for the surface of such materials. Braze alloys based on titanium-containing systems (Cu-Sn-Ti, Ag-Cu-Ti, In-Ti) for joining and metalization of perovskite BaTiO3 ceramic were used for creating uniform coatings and strong brazed samples — Fig. 6A. The shear strength of brazed ceramic/ceramic butt joints was measured (Table 2, Figs. 7, 8). It is shown that the strength of brazed perovskite samples obtained using Cu-Sn-Ti alloys equaled 42 MPa. It is about 80% of the average strength of monolithic samples.

Beside basic requirements (particularly sufficiently high wetting) for materials to be joined, compliance of their coefficients for thermal expansion is important, because stresses caused by
thermal expansion mismatch can considerably lower the strength of the joint. Applying indium-based alloys having high plasticity is especially expedient for metalization and joining BaTiO₃ ceramics.

Additionally, the technology of brazing and metalization BTO was realized using capillary impregnation of low-melting braze alloys through titanium powder. For such alloy cleanliness, titanium powder was obtained from TiH dissociating into titanium and hydrogen during heating. The indium was deposition on the titanic powder layer onto a BaTiO₃ ceramics surface. During heating up to 970 K, indium spread well over the whole BaTiO₃ surface and filling brazing gap. The thin film of In-Ti is easily formed on the surface of perovskite ceramic in such conditions — Fig. 6B.

Ferroelectric BaTiO₃

Barium titanate with a stoichiometric structure having high ferro- and piezoelectric characteristics can be heated up without any changes only within a oxygen-containing environment (in air). For such materials joining, special braze alloys and technological processes are required. Oxygen being dissolved in some metals leads to a substantial increase of wetting degree and adhesion of these metals to ceramics. The oxygen effect on wetting and also on interface and surface tension of metal melts was investigated earlier in our works (Refs. 14, 25–28). It has been shown that oxygen effectively increases the adhesion of Cu, Ag, Ni, and some other metals to ionic compounds, for example, to oxides. Several systems (Cu-O-Al₂O₃, Cu-O-MgO, Ni-O-Al₂O₃, Ag-O-Al₂O₃, Ag-Cu-O-Al₂O₃) were studied in detail. The Ag-Cu-O system is especially interesting. We have made the assumption that the “oxygen” technology will work for ferroelectric barium titanate as well. According to Refs. 14, 15, oxygen that has sufficient affinity to an electron, being dissolved in liquid metal, will increase the wettability of a surface for ionic or ion-covalent substances.

Up to now, there are only solitary works concerning the possibility of a perovskite compound (Pb(Mg₀.₃₃Nb₀.₆₇)O₃) for wetting and joining by Ag-CuO alloys (Ref. 29). Scientific background of this process is not developed; the reasons for oxygen influence on wetting are not explained in this work.

Thus, the investigation of wetting ceramic ferroelectric materials based on BTO, elaboration of braze compositions and technological conditions for brazed BTO ceramic joints, and creation of strongly adherent metal coatings on the ferroelectric perovskite ceramic surfaces were the main purpose of the present work. The Ag-Cu-O system alloys were used as a braze alloys base.

Experimental Data and Discussion

For ferroelectric BTO ceramic, experiments and technological processes were carried out per method in air media and, for the first time, under pure oxygen atmosphere using the sessile drop method as well. For this purpose, a special device was created — Fig. 9. Experiments were carried out in oxygen flow with the partial pressure of oxygen about 1 atm at 1250, 1320, and 1370 K. Technical pure oxygen was used. But the oxygen is reactive. Pure oxygen at high pressure, such as from a cylinder, can react violently with common materials such as oil and grease. Take all reasonably practicable precautions to ensure safety to prevent oxygen enrichment by keeping oxygen equipment in good condition and taking care when using it. Good ventilation will also re-
duce the risk of oxygen enrichment (Refs. 15, 30).

In Fig. 10, data on BaTiO$_3$ wetting by silver and silver-copper alloy are presented.

Oxygen dissolved in Ag-Cu alloy works as a strong adhesive element. Contact angle for pure silver in vacuum at 1250 K was equal to 129 deg, the same value in air was 96 deg, and in pure oxygen –75 deg. Copper addition to silver melt (~10% (at.)) leads to considerable contact angle decrease to 45–47 deg (in air) and to almost complete spreading of alloy (θ ≈ 5–10 deg) in the pure oxygen atmosphere.

According to Ref. 14, oxygen is an adhesion active and surface active element. In the liquid metal, oxygen exists in the form of O$^2$ ions and can form complex metal-oxygen particles with metal ions in melt (Me$^{2+}$ – O$^2$). Such complex particles have a positive pole at metal ion and negative pole at oxygen ion. Positive metallic ion of the complex is adsorbed on negatively charged oxygen ions, forming the surface of BaTiO$_3$. Localization of external electrons for metal ion at oxygen ion must weaken the metallic bond intensity with other metal ions. The bond between the metallic ion of metal-oxygen complex and another metal atom must be weaker than metallic bond atoms with each other. When the bond energy complex-Me is less than bond energy Me-Me, it is the condition for metal-oxygen complex surface activity. Adsorption of metal ions on the negative charged oxide surface results in high adhesion.

The temperature increase intensifies the wetting process; contact angle decreases to 25–30 deg in air (with 10% (at.) of Cu in liquid Ag). For pure oxygen, full spreading can be reached with Cu content at about 6–7% (at.).

The high capillary activity of alloys in a pure oxygen atmosphere is caused by a high equilibrium concentration of oxygen in melt under high oxygen partial pressure (1 atm). Oxygen partial pressure for the air is 0.21 atm. Concentration of oxygen that saturates metal melt is described by Siverts’s law (in many cases) (Refs. 31, 32),

$$\left[O_2\right]_{\text{liquid metal}} = k \sqrt{p(O_2)}$$  \hspace{1cm} (2)

where $p(O_2)$ is oxygen partial pressure and $k$ is constant.

The concentration of oxygen dissolved in the silver melt in air is equal:

$$\left[O\right]_{\text{air}} = k \sqrt{0.21}$$  \hspace{1cm} (3)
Brazing of Ferroelectric Barium Titanate

Under the air, and especially under pure oxygen atmosphere, the process of joining (brazing) of ferroelectric BaTiO₃ ceramics can be performed.

Braze alloy Ag-10 Cu was used for joining BTO ceramics in air and Ag-3 Cu under pure oxygen atmosphere.

The brazing process for ferroelectric barium titanate must be carried out in the oxygen-containing environment — in air or preferably pure oxygen. For brazing and metalization of ferroelectric ceramic BaTiO₃ used Ag-Cu-O alloys. Based on obtained data, metal-oxygen technologies of metalization and brazing BaTiO₃ ceramics in air and pure oxygen atmosphere were developed, and also a method of metalization with high adhesion using metal melts containing oxygen.

The plastic In-Ti alloy was tested for joining BTO ceramics in air and Ag-3 Cu under pure oxygen atmosphere. The brazing alloy Ag-10 Cu was used for joining BTO ceramics in air and Ag-3 Cu under pure oxygen atmosphere.

The brazed and metalized ferroelectric, barium titanate samples were obtained using plastic In-Ti filler alloy in vacuum at 720 K. It was possibly because such ceramic begins to lose oxygen and ferroelectric properties in vacuum at heating above 900 K.

Conclusion

A combined investigation including contact interaction and wetting of BaTiO₃ perovskite ceramics by liquid metals was carried out. Two states of barium titanate were studied.

For semiconducting BaTiO₃ with an oxygen defect, experiments in vacuum for 13 pure metals and Ti-containing alloys (Cu-Sn-Ti, Cu-Ga-Ti, and Ag-Cu-Ti) were carried out. Most of the metals under investigation do not wet BaTiO₃. Titanium addition sharply increases capillary properties and adhesion. Compositions of capillary active braze alloys, plus methods in brazing and metalization BaTiO₃ for high contact strength achieving, were found.

References

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