

## Technology of sintering, heat-treatment process; alloy formation in a heterogeneous system under the action of an electric current.

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**Abstract:** The question of the significant increase in the level of dissolution and alloying within a heterogeneous composite around a more refractory component in the presence of a liquid phase with passage of an electric current is considered. Redistribution of a substance into the depth of molten tin under the action of an electromagnetic forces is demonstrated using the system Cu-Sn as an example.

### 1. Introduction

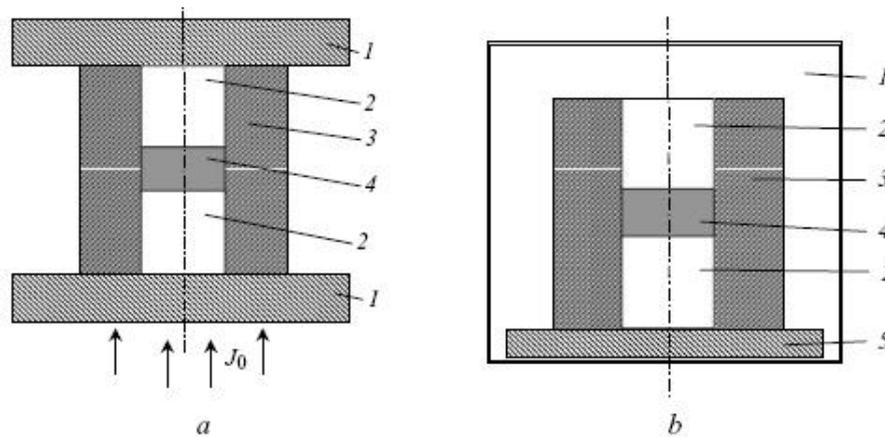
In preparing heterogeneous composite materials (CM) that have a readily-melting component in pure form (or alloys) of them as an impregnating component or solidifying matrix) conditions almost always arise that prevent total interaction at the surface of the more refractory component. With the requirement for increasing the degree of dissolution, interaction, alloy formation, or mechanical adhesion of the two phases wetting additions are introduced, the treatment temperature is increased, and various external force effects are used on the system as a whole. These actions are not always desirable since they may worsen CM operating properties.

One of the force actions supplementing or replacing normal heat treatment may be the simultaneous actions of a magnetic and electric fields with direct passage of an electric current through the CM during its manufacture or additional treatment. Here with the occurrence of a liquid phase within space around the more refractory component electroconvection arises under the action of an electric current. It promotes forced transfer of part of the diffusing element from the surface of the solid component into the surrounding material by means of liquid flow around an inclusion.

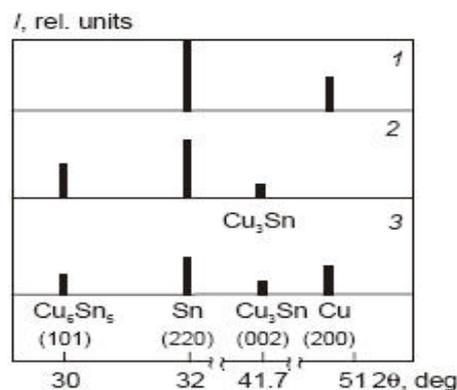
### 2. Experimental studies

Spherical particles of copper 50-55 $\mu\text{m}$  in diameter were used as the filler (solid inclusions), and tin was used as the solidifying matrix (liquid medium). The flow rate for the liquid phase (molten tin with viscosity  $\eta=1,5 \cdot 10^{-3} \text{ kg}/(\text{m} \cdot \text{sec})$  at about 350-400 $^{\circ}\text{C}$ ) at the surface of the more refractory CM component is of 3-3,5  $\mu\text{m}/\text{sec}$  with the density of an electric current passing through the CM of 100-150  $\text{A}/\text{cm}^2$ . With this flow rate transfer of diffusing element by the liquid phase should occur quite rapidly, and this certainly affects the time for total interaction within the CM body.

Theoretical calculations were checked by experiment. A composite of solid copper (20 mass%) – molten tin with the regime indicated above (treatment time about 2 min) was studied. This composite was annealed in a muffle furnace for about 1200 sec under the same temperature conditions ( $\sim 350^{\circ}\text{C}$ ), but without direct passage of an electric current. A schematic depiction of the experimental assembly is shown in Fig.1.



**Fig.1. Schematic depiction of assemblies for performing experiments in the system "solid spherical inclusions – liquid current-conducting medium". (a) With passage of an electric current: 1) press current-conducting plates; 2) electrodes-punches; 3) die made of graphite MPG-6; 4) "solid particles-molten current-conducting medium" mixture; (b) annealing in a muffle furnace: 1) furnace internal space; 2) punches; 3) die made of graphite MPG-6; 4) "solid particles-molten current-conducting medium" mixture; 5) stand.**



**Fig.2. X-ray diffraction diagram (scheme) for specimens of the Cu-Sn system prepared by different methods. (1) Initial state of copper - tin mixture; (2) after electric treatment; (3) after treatment in a muffle furnace.**

It was established by xray phase analysis that under conditions of passage of an electric current copper particles dissolve entirely in molten tin (Fig.2. 2) and the phase composition of the alloy comprises Sn with intermetallics  $\text{Cu}_6\text{Sn}_5$  and  $\text{Cu}_3\text{Sn}$ . After treatment in a muffle furnace the composite contains copper in pure form, tin, and intermetallics  $\text{Cu}_6\text{Sn}_5$  and  $\text{Cu}_3\text{Sn}$  (Fig.2. 3). This is confirmed by metallographic studies (Fig.3.).

After electric treatment the CM contains intermetallics  $\text{Cu}_6\text{Sn}_5$  and  $\text{Cu}_3\text{Sn}$  (Fig.3a.), and there is no copper in pure form in a specimen. This intermetallic material has the form of plates with a width of a 0,5-1,0  $\mu\text{m}$  and a length up to 20 $\mu\text{m}$ . These structural elements are absent from specimens treated in a muffle furnace (Fig.2.), and these CM contain copper in pure form with an intermetallic border of  $\text{Cu}_6\text{Sn}_5$  and  $\text{Cu}_3\text{Sn}$  phases (Fig.3b.).

This points to a markedly lower degree of interaction (including alloy formation) in a mixture treated in the muffle furnace. That is with the direct action of an electric current in the system mass transfer is more intense, which also promotes an increase in the degree of mass transfer from the surface of a solid inclusion into the molten conducting medium.

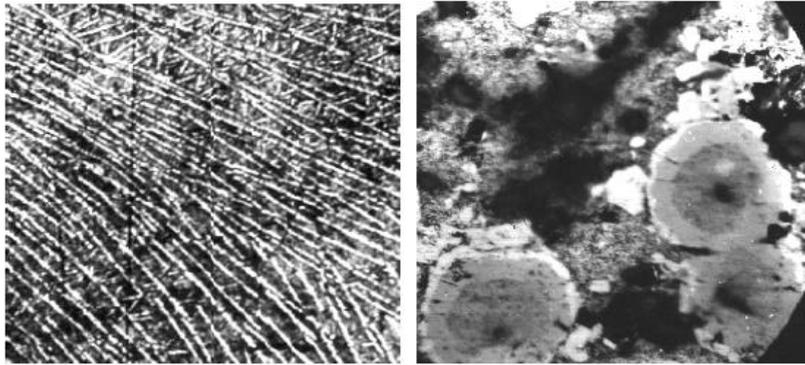


Fig.3. Microstructure of Cu-Sn composite specimens prepared with use of an electric current (a) and treatment in a muffle furnace (b). Magnification: 60 (a) and 500 (b).

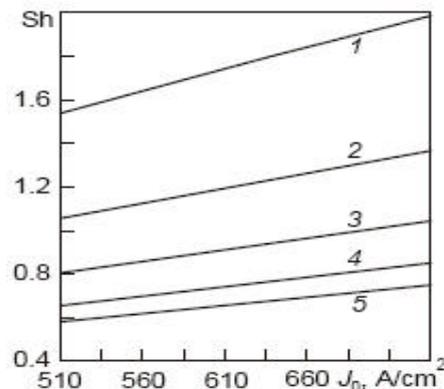
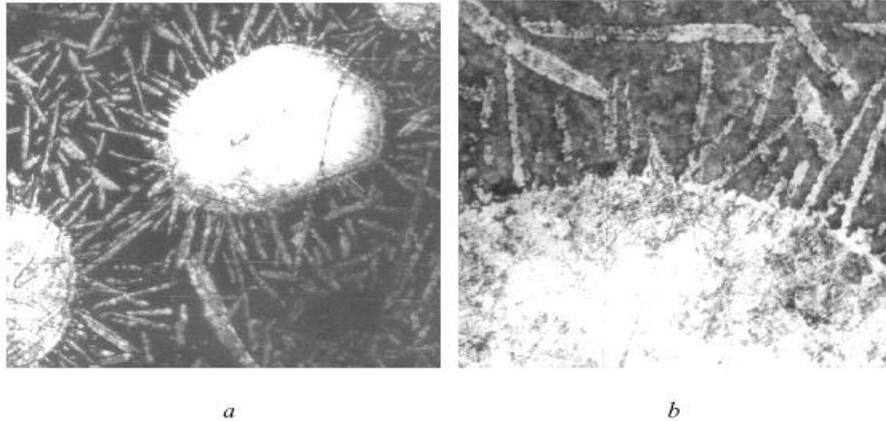


Fig.4. Sherwood number  $Sh$  as a function of electric current density  $J_0$  in a Cu-Sn composite at temperatures of 300 (1), 405 (2), 510 (3), 615 (4) and 700°C (5).

This is a basis for assuming that the mechanism of this phenomenon is electroconvection. Proceeding from the treatment time it is possible to conclude that electroconvective mass transfer exceeds by more than an order of magnitude mass transfer by the atomic diffusion mechanism. Prior calculations of the Sherwood criterion, determined here as the ratio of value of diffusion flows with electroconvective and atomic diffusion, give a value of 1,0-1,8 in the temperature range 300-400°C with an electric current density from 100 to 700 A/cm<sup>2</sup> (Fig.4.).

In practice the Sherwood number may be much greater since most probably the model presentation [2, 3, 5, 6] does not consider the activating effect of supplementary processes in the system (reaction diffusion type), and also the specific conditions for performing studies.

A similar result was also obtained in studying alloyformation in a system containing as inclusions particles with a diameter of about 0,7mm with a mass fraction of 80%. The treatment temperature was 600°-670°C, and its duration was up to 120sec. Pure tin, intermetallics  $Cu_{31}Sn_8$  and  $Cu_3Sn$  in the form of plates 0,7-5,0 $\mu$ m thick and with a length up to 100  $\mu$ m were detected in the structure of specimens. The structure and phase composition of Cu-Sn powder mixture heat treated by the traditional method with the same temperature regimes as with electric treatment were studied for comparison. Complete alloy formation occurs with sintering in the range 650-700°C for 2400-3600 sec.



**Fig.5. Microstructure of specimens of Cu-Sn composite prepared with using direct passage of an electric current for 15 sec at ~ 350°C. Magnification: 50 (a) and 200 (b).**

The composition of the specimens obtained was the same as in the first case, and intermetallic phases had a width of 5-25µm and a length of 1,6-1,7 mm. A study of the structure of specimens of this system treated electrically for a short time (15-20 sec) made it possible to determine how acceleration of alloy formation occurs in the mixture. Around a copper particle (Fig.5.) there is a thin layer of intermetallic phase  $Cu_6Sn_5$ , and there are plates of the same intermetallic that are directed into the depth of the tin. Possibly due to the effect of electromagnetic phenomena that arise with direct passage of an electric current through a CM (including the volumetric Lorentz force) part of the intermetallic phase separates from the surface and moves at rates that have a radial component.

### 3. Conclusions

Intensification of mass transfer and alloy formation in a heterogeneous specimen within which a liquid phase is present due to the use of the phenomenon of electroconvective diffusion gives primarily the possibility of shortening the duration of production treatment of CM. This makes it possible to exclude phenomena at which reinforcing inclusions (for example, diamond grains, strengthening fibres, specific fillers) do not stand high-temperature and prolonged treatment, and also are inadequately wetted by the liquid matrix. With sufficient wettability for components it is possible to prepare articles with a finer internal structure and improved properties compared with materials manufactured by traditional powder metallurgy technology.

### Bibliography

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