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SEMI-AUTOMATIC APPARATUS FOR MEASURING WETTING PROPERTIES AT HIGH TEMPERATURES

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Abstract

Determination of the physico-chemical interactions between liquid and solid substances is a key technological factor in many industrial processes in metallurgy, electronics or the aviation industry, where technological processes are based on soldering/brazing technologies. Understanding of the bonding process, reactions between materials and their dynamics enables to make research on new materials and joining technologies, as well as to optimise and compare the existing ones. The paper focuses on a wetting force measurement method and its practical implementation in a laboratory stand – an integrated platform for automatic wetting force measurement at high temperatures. As an example of using the laboratory stand, an analysis of Ag addition to Cu-based brazes, including measurement of the wetting force and the wetting angle, is presented.

Keywords: surface tension, wetting angle, wetting force, measurement system.

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1. Introduction

Research on new materials and joining technologies is one of the most important areas of interest in materials' engineering. Good knowledge of the structure of materials, their physical and chemical properties, behaviour in various conditions enables to design new materials or technologies as well as to customize the existing ones. In the latter case, they become more economical and offer a better quality. This research is also fostered by European Union directives (Restriction of Hazardous Substances 2011/65/EU; Waste Electrical and Electronic Equipment 2012/19/UE) determining the detailed requirements of use of certain substances in the electrical and electronic devices, which are in operation in many countries. It also forces changes in industrial technologies [1, 2]. Current research on the modern materials and their industrial applications involves development of new measurement methods providing accurate, quantitative information on the behaviour of a material in various technological processes. Modification of a material structure has to meet the requirements regarding specified properties of new materials in appropriate usage conditions. Frequently, the difficulty lies in application of a new technology in the production process, where elements made of modern materials are assembled with those of traditional ones.

Determination of the physico-chemical interactions between liquid and solid substances is a key technological factor in many industrial processes in metallurgy, electronics or the aviation industry. The primary phenomenon used in the bonding process is wetting the joined surfaces with a liquid metal, which is described by the primary interfacial impact parameters, *i.e.* the wetting force and the wetting angle [3–7]. Information on the values and dynamics of the above-mentioned parameters can be obtained by performing experiments based on the immersion method. Knowledge of the wetting dynamics enables to customize the existing

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technologies, optimize them or fit to appropriate process conditions and requirements (significant reduction of the process time and temperature, use of a protection atmosphere, application of fluxes, surface preparation, and the like). In addition, it enables to study materials and joining technologies based on soldering.

2. General concept of measurement and analysis of wetting force

A wetting force measurement procedure, consisting in observation of a specimen's weight changes during an experiment of immersing a specimen in a liquid braze, is known as the Wilhelmy plate method. A high-precision scales system measures the resultant forces acting on the vertical specimen. Based on analysis of the distribution of the forces acting on a specimen before and after the immersion (Fig. 1), the capillary wetting force is denoted as [3–7]:

$$F_c = F_{m2} - F_{m1} + S_a \rho g h \tag{1}$$

and the wetting angle:

$$\theta = \arccos\left(\frac{F_c}{P_a \sigma_{LV}}\right), \tag{2}$$

where: F_c – the capillary wetting force; F_{m1}, F_{m2} – the forces registered by the scales system before and after immersion, respectively; S_a – the cross-sectional area; ρ – the solder density; g – the gravity acceleration; h – the immersion depth; P_a – the specimen perimeter; σ_{LV} – the solder surface tension.

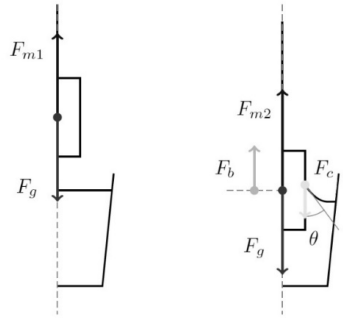

Fig. 1. Distribution of the forces acting on a specimen before and after its immersion in a fluid braze.

Equation (2) applied to a specific liquid-solid system describes a single wetting angle. In fact, there are many metastable interstates on the solid-liquid boundary arising from the material heterogeneity, impurities and surface structure modification, which affect differences of the wetting angle relative to the aforementioned angle value. When the phase boundary is moving, instead of static contact angles, one should measure dynamic contact angles, and thus determine the range of angles referred to the advancing and receding angles, which creates the wetting angle hysteresis [8].

3. Integrated platform for automatic wetting force measurement at high temperatures

3.1. Computer system overview

The integrated platform for automatic wetting force measurement at high temperatures is an autonomous stand enabling complex research on the dynamic brazing process properties –

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the wetting force at temperatures of up to 1000°C with various technological gas atmospheres. An overview of the measurement stand and its most important subsystems – heating, driver and loading systems, is presented in Fig. 2 [9].

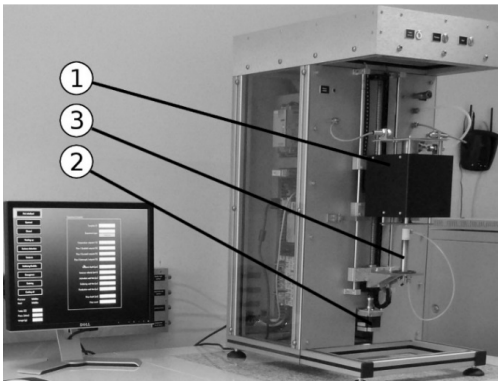

Fig. 2. An overview of the wetting force measurement system (1 – heating; 2 – driver; 3 – loading subsystems).

The system offers a wide range of working temperatures up to 1000°C with a braze bath temperature control accuracy of 0.5°C and a precisely controlled gas protective/reductive atmosphere (usually based on argon, nitrogen and hydrogen of 5N purity). The measurement cycle is fully automated, the process parameters and experiment sequence are controlled by a real-time automated system. The measurement procedure is based on analysis of the forces acting on a specimen during its immersion in a liquid braze. The integrated platform is a unique solution for industrial and laboratory purposes enabling to make research on design of new materials and soldering/brazing technologies or to optimize and verify the existing ones [9]. The commercial solutions available on the market (*e.g.* Metronelec Menisco products) offer wetting force measurements in a gas-protective atmosphere mostly at a temperature of only up to 450°C [10]. The measurement system presented in this paper enables to examine the dynamic brazing process properties at up to 1000°C.

3.2. System architecture

The architecture of the integrated platform for automatic wetting force measurement at high temperatures consists of functional blocks responsible for executing separated tasks. The basic subsystems include: heating, gas, scales and loading/drive subsystems, all supervised by an industrial software *programmable logic controller* (PLC) equipped with extension cards necessary to communicate with industrial parts/devices. An additional component of the research stand is an autonomous information system enabling to analyse the experiment results. The architecture of the research stand is presented in Fig. 3 [9].

The main part of the measurement system is a custom-built heating system based on a cylindrical resistance furnace (Fig. 4, A) enabling to heat a specimen up to 1000°C with a temperature control accuracy of 0.5°C. The heating system is controlled by a PLC software coupled with an appropriate power driver and a thermocouple receiving temperature from the furnace chamber. During the experiment, the pot with a braze material is placed in the centre of the furnace, on an appropriate base (Fig. 4, B). Location of the pot in the furnace and movement of the whole heating system in the direction of the fixed specimen are executed by a custom-built driver system (Fig. 4, G) controlled also by the PLC controller. It ensures

a position control accuracy of 0.05 mm and a speed of up to 200 mm s^{-1}. The specimen is detected at the furnace entrance by the optical detection system (Fig. 3, D) using Pepperl+Fuchs photoelectric sensors [11]. The contact between the specimen and the liquid braze is recognized by rapid specimen weight changes when the specimen face touches the solder surface. The balance subsystem is based on a Mettler Toledo weighing module with the maximum load of 220 g and accuracy of 0.1 mg [12]. During the heating some additional phenomena, *e.g.* oxidation, can occur. Therefore, the measurement system is equipped with a gas-protective atmosphere based on nitrogen (Fig. 4, E). A hydrogen and argon mixture is applied as a reduction atmosphere in the furnace chamber. The gas flow is controlled by the Brooks *mass flow controller* (MFC) elements calibrated for specified external gas sources with the maximum flow of 250 mln min^{-1} and control accuracy below 1% of the actual flow (20–100% of full scale) [13]. All components, including sensors, motors and controllers, are managed by a WAGO software PLC based on an x86 family processor, Linux operating system and CodeSys real-time environment. The PLC unit is equipped with extension cards including digital/nalog inputs/outputs, a stepper motor controller, RS interfaces and others [14].

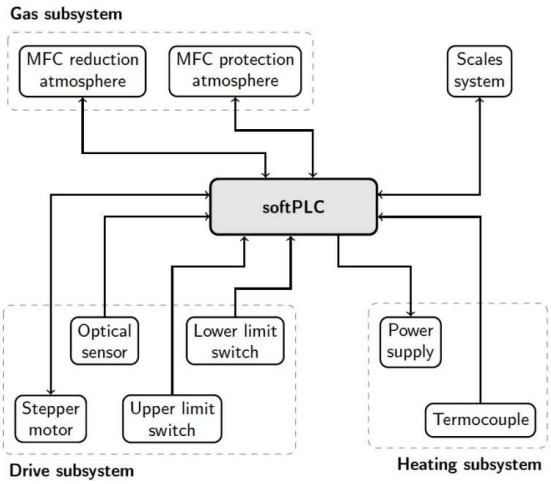

Fig. 3. The architecture of the research stand with reference to heating, gas, drive, scales subsystems and controlling devices.

3.3. Flow diagram of wettability measurement experiment

The sequence of tasks executed by all hardware system components according to the process parameters set by the user, which present the required values of the main parameters of the process, *i.e.* the temperature, the gas flows, *etc*., is illustrated in Fig. 5. Selected process parameters that can be set by the user are presented in Table 1. The experiment tasks refer to the appropriate states of the device and define all actions and conditions necessary to proceed to the next experiment step. The experiments can differ in details; however, their general process outline is similar. During the experiment, the current process variables (*i.e*. weight, temperature, specimen position, time) are recorded.

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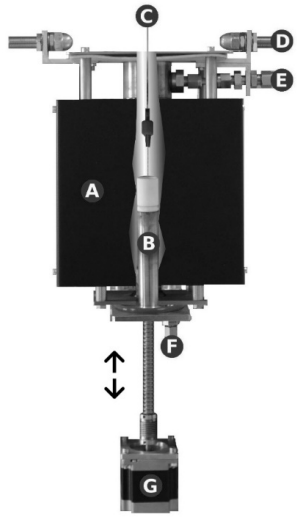

Fig. 4. A cross-section of the measurement system (A – the heating system; B – the base and the pot with a liquid braze; C – the tested specimen and its mounting; D – the specimen detection system; E, F – the gas supply system; G – the driver system).

Table 1. Selected process parameters that can be set by the user.

Parameter	Description
Temperature [°C]	Process temperature (up to 1000 °C)
Immersion depth [mm]	Specimen immersion depth in fluid solder / braze
Immersion speed [mm s^{-1}]	Specimen immersion speed
Emergence speed [mm s^{-1}]	Specimen removal speed
Step depth [mm]	Specimen immersion step depth in fluid solder / braze (multiple immersion steps' experiment)
Step count	Specimen immersion step count (multiple immersion steps' experiment)
Activation time [s]	Time of specimen stay in the furnace interior, over a solder / braze bath, before specimen immersion
Stabilization time [s]	Time of specimen stay in fluid solder / braze
Cooling time [s]	Time of specimen stay in the furnace interior, over a solder / braze bath, after specimen removal

The experiment is carried out at a high temperature, in the presence of a protective gas atmosphere. Stabilization of the thermal conditions and gas reduction atmosphere before the main part of the experiment is vital. The experiment procedure starts with a vertical movement of the furnace with the pot filled with a braze towards the specimen – until the specimen is detected at the furnace entrance by the optical sensor (Fig. 7, pos. A). After the detection, the movement still continues, until the specimen is placed in the furnace chamber at a fixed position to activate the specimen surface for a required time. Next, the furnace vertical movement towards the specimen continues until the contact with the braze surface is detected (Fig. 7, pos. B). The contact is detected by the scales system – when the face of the specimen reaches the braze bath, rapid weight changes are observed. After the contact detection, the specimen is placed at an appropriate depth, where it stays for a required stabilization time (Fig. 7, pos. D–F). Finally, after the specimen is taken out of the braze and kept above its surface for

an appropriate time, the specimen is removed from the furnace, at which point the experiment ends [9].

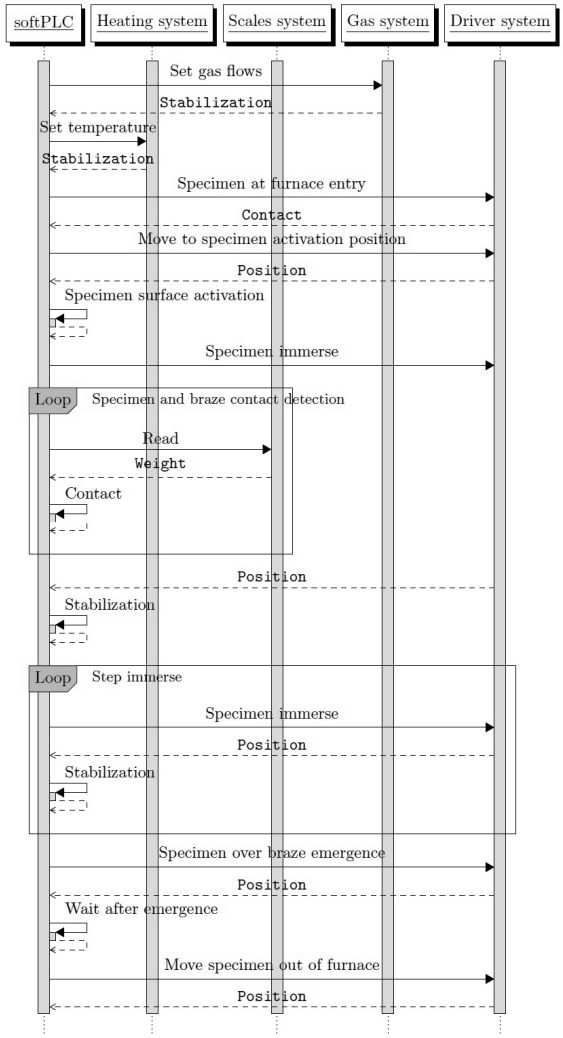

Fig. 5. A flow diagram of the measurement experiment.

The apparatus, due to a wide range of process parameters that can be set by the user, offers a great flexibility in research planning. The proper selection of parameters affects the correctness and purpose of executed experiment. The most important parameters determining the type of study are timing parameters (immersion and emergence speed, stabilization time) enabling to focus on measurement of the wetting dynamics (*e.g.* advancing and receding angles) or observation of the wetting changes during the specimen's static stay in a fluid braze. The correct determination of most available parameters requires sufficient experience from the operator, and it should be preceded with execution of a series of testing experiments. For example, the time of the specimen's stay in a fluid braze should match, in most cases, the stabilization of the wetting force. Some of the parameters, even those fundamental to the

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process, such as temperature or gas flows, can be verified after the experiment with an additional study, *e.g.* microstructure research on oxides or on the existence of specific phases. A number of technical parameters are not available to the user, because they are related to the activity and protection of the research stand (component devices reading frequency, speed and position limits, *etc.*), but they should be taken into account when planning the experiment flow and other parameters.

4. Example of analysis of Ag addition to Cu-based brazes

As an example of using the integrated platform for automatic wetting force measurement at high temperatures, an analysis of Ag addition to Cu-based brazes was made. The immersion experiments for the Cu specimens and Cu-based brazes with Ag addition, commonly used in electrotechnical and mechanical applications, were carried out for three different brazes (braze 1–93.8% Cu, 6.2% P; braze 2–91.8% Cu, 6.2% P, 2% Ag; braze 3–80% Cu, 5% P, 15% Ag). Parameters of the experiments are shown in Table 2. The wettability changes during the immersion process of copper specimens in liquid brazes are presented in Fig. 6.

Table 2. Parameters of the immersion experiments.

Parameter	Value	Parameter	Value
Temperatures [°C]	740–760	Activation time [s]	20
Immersion depth [mm]	5	Stabilization time [s]	5
Immersion speed [mm s^{-1}]	5	Cooling time [s]	10

A detailed analysis of the wetting force changes during the specimen's static stay in a fluid braze is presented in Fig. 7. The main stage of the experiment starts with contacting the specimen front surface with the liquid braze (Fig. 7, pos. B). During the specimen sinking process, the acting buoyancy force rises linearly with an immersion depth. At the same time, the braze bath surface is deflected and a down-curved meniscus is formed. The wetting angle θ is changing until it reaches its maximum value and forms an obtuse angle (Fig. 7, pos. C). The temperature of the specimen rises to the braze temperature. Bonds between the two-phase atoms and the SL-phase boundary are created. The C–F stages correspond to the wetting progress. The capillary force is increasing, with its value equal to the buoyancy force at point D and with a value equal to 0 at point E, where the wetting angle reaches 90°. From point E, the wetting angle forms an acute angle towards the metastable equilibrium value θ^0, where the resultant force acting on the specimen remains in balance. At point F, the wetting force reaches 90% of its maximum value, which is needed to create a joint with good properties. The last stage is the emergence process (Fig. 7, pos. G). The liquid meniscus is broken off and the experiment is ended (Fig. 7, pos. H). The dynamic parameters of the tested braze materials are shown in Table 3. Additionally, research on the surface tension at a required temperature was performed for each braze material using the lying drop method implemented in the ThermoWet device [15]. Knowledge of the surface tension parameter is needed to calculate the wetting angle changes as a function of time (2). The wetting angle changes registered for Cu-based brazes with Ag addition are presented in Fig. 8.

The immersion experiments were carried out for Cu specimens and three Cu-based brazes specified above. The experiment results prove that addition of Ag in the composition of brazes affects the wetting dynamics and parameters of braze materials and process conditions. The addition of Ag in a braze composition causes reduction of the wetting force – for braze 1 the registered wetting force is about 7.84 mN and the metastable equilibrium wetting angle is about 58°, for braze 3 the force value is 4.67 mN and the required wetting angle 72°. For brazes with

a lower amount of Ag addition, the wetting dynamics is better – for braze 1 and braze 2 the calculated dynamics ratio is about 0.3, but for braze 3 it exceeds 0.4. The dynamics ratio is a basic parameter for technology design which specifies the speed of the wetting process (a lower value of the ratio denotes a rectangular shape of the wetting curve for rapid wetting, while a value close to 1 indicates a slow increase in the wetting force). However, increasing the Ag component lowers the process temperature (the recommended operating temperature for braze 1 is 760°C, but for braze 3–730°C) [16].

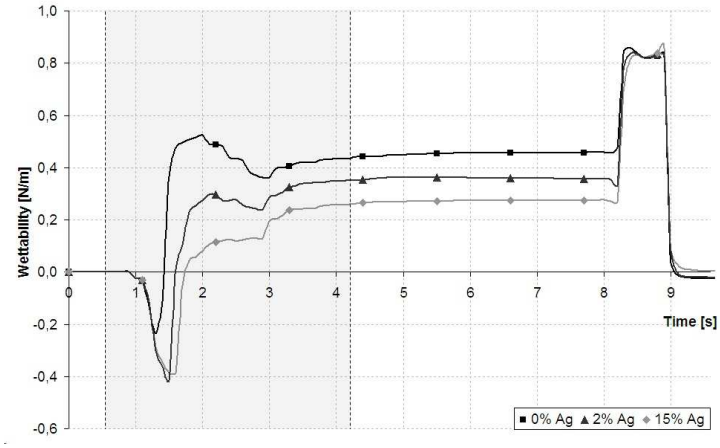

Fig. 6. The wettability changes registered for Cu-based brazes with Ag addition described above (braze 1 – ■; braze 2 – ▲; braze 3 – ♦) with an area selected for a detailed analysis of dynamics.

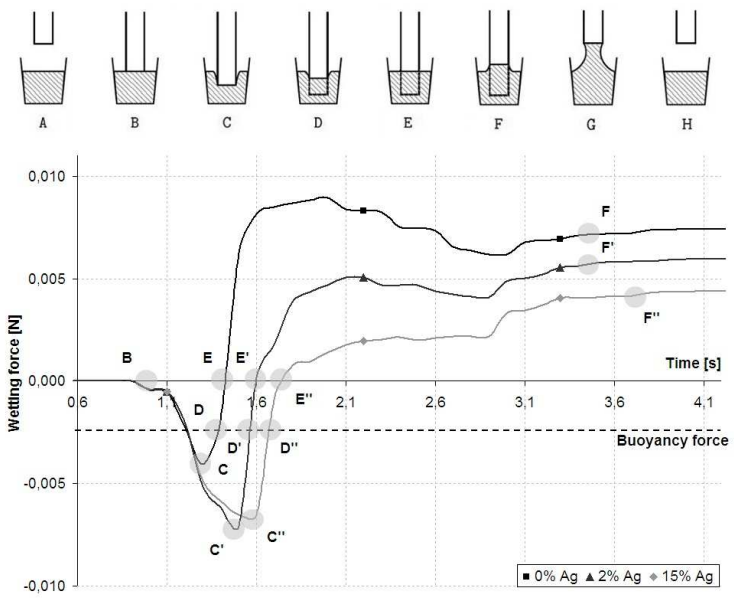

Fig. 7. The wetting force changes and specific points for Cu-based brazes with Ag addition (braze 1 – ■; braze 2 – ▲; braze 3 – ♦) within the marked area (Fig. 6).

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Table 3. The wetting dynamics parameters of the immersion experiments for Cu flat specimens and Cu-based brazes with Ag addition.

Parameter	Braze 1	Braze 2	Braze 3
t_0 [s], point B	1.0	1.0	1.0
$t_{\theta max}$, point C	1.3	1.4	1.6
t_{FB}, point D	1.4	1.5	1.7
$t_{\theta = 90°}$, point E	1.5	1.6	1.8
$t_{90\%}$ [s], point F	3.4	3.3	3.8
t_C [s]	7.8	7.8	7.8
F_G [mN]	7.84	6.0	4.67
$(t_{90\%} - t_0) / (t_C - t_0)$	0.35	0.34	0.41

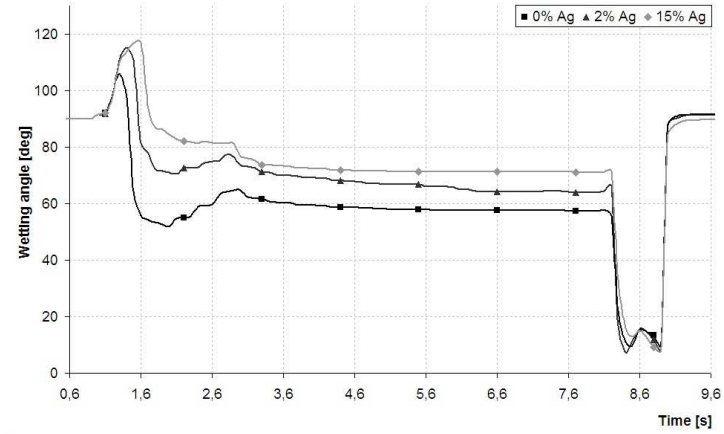

Fig. 8. The wetting angle changes registered for Cu-based brazes with Ag addition (braze 1 – ■; braze 2 – ▲; braze 3 – ♦).

5. Conclusions

Determination of conditions for the production of good quality material joints is nowadays carried out by means of equilibrium wettability tests. In the paper, a methodology of wetting force measurement, including a general concept of the immersion experiment and calculation of its parameters, is presented. The methodology was used in the automatic research stand – an integrated platform for automatic determination of high-temperature braze wettability, enabling to perform a comprehensive study of dynamic properties of brazes at temperatures of up to 1000°C with the use of various technological gas atmospheres. The research carried out on an integrated platform supplemented with an additional analysis of the microstructure provides comprehensive information about material properties and their behaviour that can be used for industrial and laboratory purposes regarding the design of new joining technologies and materials, optimization of brazing and soldering process parameters as well as verification of quality of the existing technologies.

In the paper, an example of research on Ag addition to Cu-based brazes is presented. The immersion experiments carried out for Cu specimens and Cu-based brazes (braze 1–93.8% Cu, 6.2% P; braze 2–91.8% Cu, 6.2% P, 2% Ag; braze 3–80% Cu, 5% P, 15% Ag) prove that addition of Ag in the composition of brazes affects the wetting dynamics and parameters

of braze materials and process conditions. The addition of Ag in a braze composition causes a reduction of the wetting force and a slowdown of the wetting dynamics. On the other hand, increasing the Ag component lowers the process temperature.

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