

PERNER'S CONTACTS



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IMPROVEMENT OF SESSILE DROP METHOD FOR THE WETTING ANGLE DETERMINATION

József Hlinka¹, Zoltán Weltsch², János Berzy, Attila Szmejkál

Summary: The wetting angle (θ) of molten Ag drops on graphite substrate is investigated. The effect of oxygen on the wetting ability of liquid metals has a huge importance. The oxide covered surface of the substrate deteriorates wetting. A new insulation of the furnace was inaugurated, which can reduce the oxygen concentration. Automatic contact angle measuring technique was applied, which analyze the recorded image from the drop, and determine the wetting angle without any dispersion of evaluation.

Key words: wetting angle, sessile drop method, temperature dependence, silver.

INTRODUCTION

The significance of Ag-based alloys is important in brazing and welding processes (1). The wetting phenomena between the filling alloy melt (brazing alloy) and the base material have a central role in obtaining sufficient quality, which serve the formation of steady joint between the parts. Alloying elements are also often used for this purpose in order to lower the melting point and also for increasing the wettability between the parts to be fixed. Both properties depend on the individual properties of alloying elements, including their surface activity in the Ag host and also their interaction with oxygen, which cannot be fully eliminated from the ambient atmosphere.

1. EXPERIMENTS: DEVICES AND MATERIALS

Detailed description of the measuring system and the sample preparation (alloys and substrates qualification) were reported in earlier papers (2, 3).

1.1 The insulation of the furnace

An efficient around the furnace block is necessary because the sealing of the vacuum chamber does not tolerate the high temperature which is created by the resistance heating during heating up the sample. The insulation increases the size of the internal surfaces. However, the bigger inner surface in the vacuum chamber causes bigger irremovable oxygen concentration.

The effect of oxygen on the wetting ability of liquid metals has already been reported in a large number of investigations. Reports show that the oxygen in the metals acts as a surface-active element. The surfaces of oxides are generally thought to be covered with oxygen.

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When a liquid metal comes into contact with the surface, it is thought that the affinity between the metal and the oxygen affects the wettability. This affinity between liquid metal and oxygen is evaluated via measuring the strength of bonding between the surface of the liquid metal and solid oxide. It is thought to involve a physical interaction as well as chemical interaction. The oxide surface on the substrate deteriorate wetting.

The construction, which can solve the problem, is shown in Figure 1. The quartz glass is smooth enough, so the increase of the surface is as small as possible. The essence of construction is a double-wall cryostat body, which can be evacuated. This cryostat body ensure excellent heat isolation around the resistance furnace. An insulating blanket of Al_2O_3 layer is placed into the quartz glass cylinder. The quartz glass resists sudden heat shocks and its poor heat conductivity makes it suitable for the purpose. Cooling water circulates around the insulation, which cools the environment. The insulation concentrates and reduces the spread of heat produced by the resistance heating.

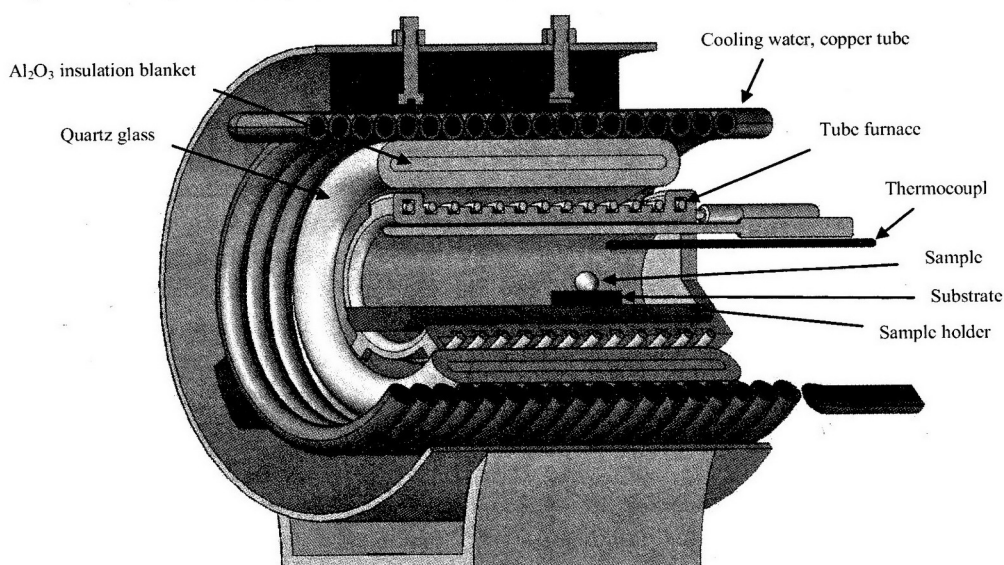


Fig. 1 – Furnace and insulation

1.2 Automation of wetting angle measurement

It is important to ensure invariant conditions during the wetting angle measurement allowing us comparing different measurement results. This aim was kept in mind during the investigation processes of both the measurement and evaluation part of experiments.

In case of sessile drop method we take a digital photo about the molten drop, we analyze it and determine the wetting angle. The image must show the contour of the drop sharply and noiselessly.

Earlier, the evaluation was performed manually, step by step: The picture was scanned with a program automatically, and then the 2 corner points of the drop (contact points with the substrate) were selected manually. These points define the top of the substrate as a straight

line. Next step was to draw two tangent lines to the drop contour from the two contact points. The wetting angles are the drop-side angles enclosed by the initially defined line and the tangent lines. The program prints to screen the results, and saves them to a txt file. The problem of this evaluation mode is the human uncertainty. The results can be different depending on the measuring person and tests. Even with perfect pictures deviations were discovered in the measuring results. The reason is that the initially selected sideward contact points, thereby the horizontal line and the tangents depend on the accuracy of the measuring person.

The automation is the solution for the elimination of deviations. The automatic evaluation program firstly converts the colourful measurement picture into a black and white image (Fig. 2). Both the substrate and the drop will be white and the environment will be black, so the outline of the drop can be determined easier. In the next step the program starts vertical search at a specified interval from the right and left sides of the image to find the first transition points between the black and white areas. Then, the program determines the focal points of these sets of transition points on both sides. The connection line of these focal points represents the top of the substrate (see Fig. 2).

Subsequently, it starts horizontal search at a specified interval and height above the previous line to find the first transition points between the black and white areas from the right and left sides of the image. In this way two sets of points are reached (curved scatter graphs on Fig. 2) representing the contour of the drop. The program fits two circles on these point sets. Solving the geometrical equations of the line and the two circles the program obtains the intersection points of circles and the base line (see Fig. 2). Choosing the two appropriate solutions, we get the two sidewise contact points of the drop. The radiuses – connecting the intersection points and the centres of the circles – are perpendiculars to the tangents that can be drawn to the circles at the intersection points. Thus, wetting angles can be got from the angles of the base line and the radiuses via coordinate-geometrical calculations.

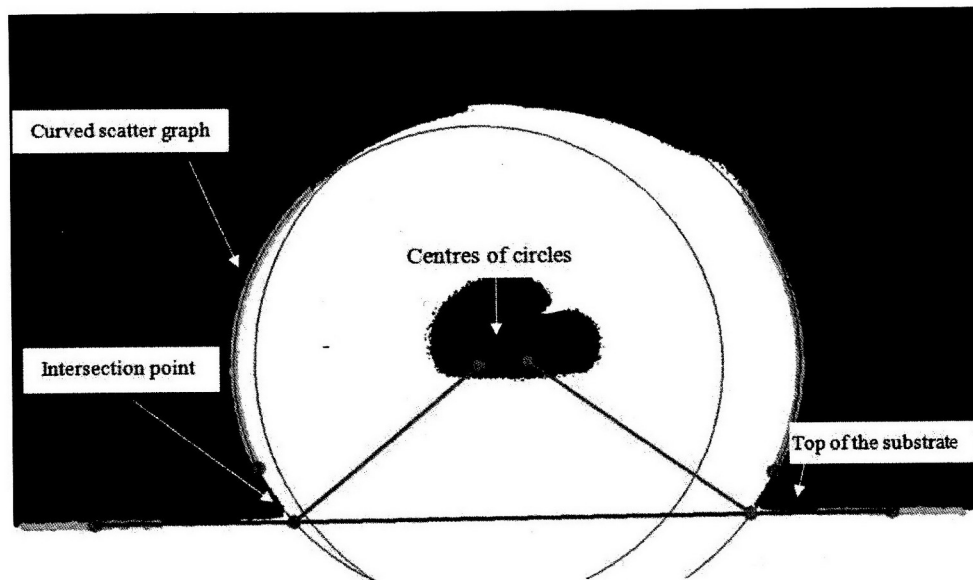


Fig. 2 – Molten pure Ag drop on graphite substrate

With this method the inaccuracy of human work during the manual evaluation can be eliminated. The evaluation of the experiment results is executed beside the same conditions, so the comparison of the outcomes is reliable, reflecting the real correlations.

1.3 Method of alloy preparation

Alloys were prepared from high purity (4N) components, using high frequency induction melting in cold crucible under inert (Ar) atmosphere. The graphite substrate was made from high purity, porosity free base material. Inert (Ar) atmosphere was applied during the alloy preparation and the wetting experiments.

The material is placed in a crucible consisting of water-cooled copper segments. The crucible is placed into an induction coil. This method, also known as levitation melting, is used to develop very special alloys that are free from contamination due to the absence of contact with the crucible. The set-up of the device is shown in Figure 3.

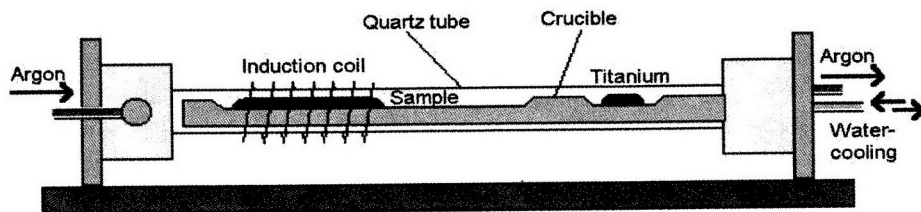


Fig. 3 – Experimental arrangements for alloy sample preparation

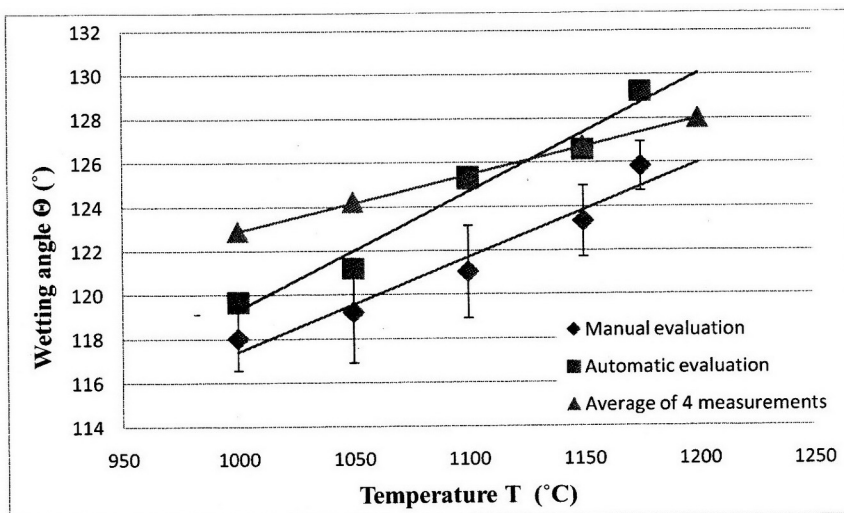


Fig. 5 – Wetting angles of pure Ag on graphite substrate

2.3 Measurement of the surface roughness of substrates

The surface roughness of the substrates has a profound and technologically important influence on the behaviour of wetting fluids. In one of the first studies the effects of surface roughness on the equilibrium contact angle (Θ_0 between wetting liquid and solid substrate) was reported by Wenzel [4]. He claimed that surfaces that wet ($\Theta_0 < 90^\circ$) in smooth state will wet even better in rough state. Conversely, argued that if the smooth surface does not wet well, the rough surface wets even worse. This behaviour is expressed in the relationship

$$\cos \Theta_r = r \cdot \cos \Theta_0, \quad (1)$$

where Θ_r is the contact angle on a surface with a roughness ratio r , given by the ratio of actual wetted surface area to geometric or projected area. According to (Eq. 1) equation, the rise of the roughness of a surface decreases the contact angle if $\Theta_0 < 90^\circ$, whereas it increases the contact angle if $\Theta_0 > 90^\circ$.

The surface roughness was determined using a 3D laser profilometer (Rodestock RM600 surface topography measurement system). Roughness parameters, which are presented here, are: R_a , the arithmetical mean line to a roughness profile; λ , the main horizontal wavelength; r , defined as the ratio of the true area of the solid surface to the apparent area. The tested area was 10^{-4} m^2 and the roughness measurement was repeated 5 times. The measured roughness parameters are shown in Table 1 and Fig 5.

Tab. 1 - Roughness parameters of substrates

substrate	R_a	λ	r
C	0.235 ±0.006	14.58 ±0.34	1.009 ±0.0005

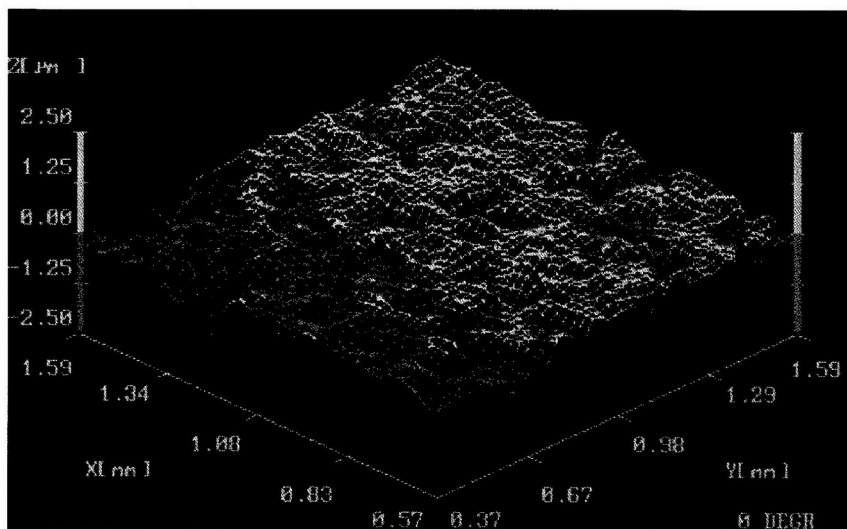


Fig. 5 – Graphite substrate surface roughness

CONCLUSION

The wetting behaviour of pure Ag in contact with a graphite substrate was analyzed. A small decrease in the contact angle can be detected with the newly developed equipment, which ensures oxygen-free atmosphere. It means that Ag drop moistens better the graphite substrate in oxide-free atmosphere. It can be concluded that using this equipment we can focus better on the influence of alloying effect on wetting ability during subsequent measurements.

The drop profile of the sample was acquired using a CCD camera aligned in the plane of the substrate. The images were processed by self-made evaluation software in a Matlab environment. This software allowed a very fast, automatic determination of the drop profile and the evaluation of surface tension and contact angles.

Combining the measurements made in oxygen free atmosphere with the automatic contact angle determination, we got a system with minimal error of measurement. Thus, the results of further measurements can be used for an accurate comparison of the wetting properties of different samples.

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