The methodological specificity of measuring surface tension by the methods of large drop and maximum pressure in a drop

R Kh Dadashev¹², R A Kutuev¹ and V A Sozaev²

¹ Faculty of Physics and Mathematics. Chechen State University, Grozny, Russia
² North Caucasus Mining and Metallurgical Institute (State Technological University), Vladikavkaz, Russia,
³ Materials Issues Science Center Academy of Sciences of the Chechen Republic, Grozny, Russia.

E-mail: kra-07@mail.ru

Abstract. A comparative analysis of the experimental data on the surface tension (ST) of low-melting metals, obtained by the method of maximum pressure in a drop and the method of a large drop, shows that the values of ST obtained by the large drop method for all studied metals are noticeably higher than those obtained by the method of maximum pressure in a drop. The reasons for such discrepancies, although in some cases they exceed the total errors of the ST measurement by the indicated methods, have not yet been clarified. In this regard, the paper gives a comparative analysis of the methodological specificity of measuring the surface tension by the methods of maximum pressure in a gas bubble or a drop and a lying or “large” drop. It is shown that these methods are theoretically substantiated, technically perfect, have a sufficiently high accuracy and are relatively simple. Based on the identification and critical analysis of the influence of the main factors on the measured value of surface tension, the main reasons for the discrepancy between the experimental data on the surface tension of low-melting metals obtained by different methods are determined. It is shown that the underestimated ST values obtained by the method of maximum pressure in a droplet are due to the fact that all factors influencing the measured value of surface tension are directed towards a decrease in its value. Although the influence of each of these factors separately is insignificant, they together lead to a noticeable decrease in the value of surface tension. It is shown in the work that the method of maximum pressure in a drop, being the most accurate method for measuring ST, gives somewhat underestimated ST values for all low-melting metals.

1. Introduction

The method of maximum pressure in a drop and the method of lying “large” drop are among the most accurate and reliable methods for measuring the ST of metals and alloys, each of these methods having their own advantages and disadvantages. However, as it will be shown below, a comparative analysis of the experimental data on the temperature dependence of the ST of pure metals obtained by these methods shows that they differ markedly from each other, and these discrepancies for some metals go even beyond the total error in measuring the ST. It should be noted that the ST values of the studied metals, obtained by the “large” drop method, always numerically exceed the results of ST measurements by the maximum pressure in a drop method [1–4], which indicates that these
discrepancies are not associated with the influence of a random error, and are due to factors, the direction of which is the same. The nature of these factors and the reason for such discrepancies has not yet been clarified and, in our opinion, the analysis of the methodological features of measuring the surface tension by both methods in order to identify the degree of influence of each of the random factors on the ST measurement error is of a certain scientific and practical interest. Unfortunately, it is not customary to write in detail about the small details of conducting experimental research. In practice, these seemingly insignificant details can have a significant impact on the final results. In this regard, let us consider in more detail the methodological features of measuring ST by the method of maximum pressure in a drop and the influence of each of the factors leading to an error in measuring ST.

2. Methods
Until now, the method of maximum pressure in a drop, after the development of the gravitational technique by P.P. Pugachevich, remains unsurpassed in terms of the accuracy and reliability of the obtained experimental data. The widespread use of this method for measuring the ST of low-melting metals and their alloys was facilitated by devices designed by P.P. Pugachevich, N.L. Pokrovsky, V.V. Lazarev [5, 6]. This technique was appreciably improved in terms of increased productivity of experiments, while maintaining the achieved accuracy in the works of Kh.I. Ibragimov and his students [7]. They designed and tested many combined devices for the joint study of the temperature and concentration dependence of the ST and the density of metals and alloys, which took into account the specific features of the objects under study (volatility, oxidizability, experimental performance, etc.). These devices are widely used in industrial and scientific laboratories. They were demonstrated at the USSR Exhibition of Economic Achievements and two of them received bronze medals of the exhibition.

The most important advantages of gravitational devices are: the purity of the experiments, the possibility of multiple repetitions of measurements, as well as the preparation and study of the surface tension of a large number of alloys of various compositions; the possibility of unlimited mixing of the melt; relatively short measurement time; the possibility of achieving thermodynamic equilibrium of liquid with vapor before measurements. The listed advantages allow the experimenter to achieve high measurement accuracy.

The method of maximum pressure in a drop has now been brought to perfection and, along with the method of a lying drop, is widely used in practice as one of the most accurate methods for measuring the ST. However, this method has one fundamentally important drawback: it is implemented in glass devices, which imposes restrictions on the upper limit of the temperature range of surface tension measurements. Therefore, these devices can be used to study the ST of liquids up to the glass softening temperature (below 800 K). At the same time, it is not superfluous to note once again that the devices, designed on the basis of the gravitational technique of P.P. Pugachevich, to this day remain unsurpassed in accuracy and reliability.

While yielding to the gravitational technique of P.P. Pugachevich in terms of measurement accuracy, the large drop method has a number of advantages, the main of which is a significantly wide temperature range of measurements and the possibility of measuring the ST of liquids in a supercooled state [8]. On the other hand, the large drop method, in contrast to the gravitational technique, makes it possible to measure the ST even when the substrate material is wetted with a liquid.

One more fundamental advantage of the large drop method is that it has been has been significantly improved using modern information technologies [9–11]. It is them that in the recent years enabled the production of scientific devices based on this method that allow for high-precision studies of the surface properties of liquids and solutions. In particular, the KRUSS company produces unique instruments for studying the ST, contact angle and density of liquids by the lying drop method. Let us consider separately the influence of various factors on the ST value of the investigated liquid and the method for measuring ST by these methods.
3. Theory. Calculation

3.1 Peculiarities of measuring ST by the method of the lying “large” drop

The large drop method has found wide application in the study of surface properties (ST, contact angle) and the density of substances in a liquid state. Suffice it to say that this method was used to determine about 40% of the density values of substances in the liquid state [12].

In the large drop method, the ST value is determined from the shape of the liquid drop. The method is based on solving the Laplace differential equation.

\[
\frac{1}{r_1} + \frac{1}{r_2} = \frac{z}{a^2}
\]

(1)

\[
a = \sqrt[3]{\frac{\pi \sigma}{g(\rho^2 - \rho_f^2)}}
\]

(2)

is the capillary term

For a curved surface, in the special case when the surface \( z = f(x,y) \) deviates slightly from the plane \( z = 0 \). This can be expressed as

\[
\frac{\partial^2 z}{\partial x^2} + \frac{\partial^2 z}{\partial y^2} = \frac{z}{a^2}
\]

(3)

The second order-equation (3) in partial derivatives is solved approximately. It should be noted that it can be solved by quadratures, but only for some elementary forms. Equation (3) is used in various methods for determining the ST of liquids.

F. Bashfort and J. Adams [13] were the first to carry out numerical integration for the Laplace equation (3). At present, this equation is being solved with high accuracy by the methods of modern information technology.

A critical analysis of the main methods for measuring the surface tension of liquids is given in [1, 3]. Therefore, we do not dwell on the analysis of other methods for measuring surface tension, and note that the advantages of the lying (large) drop method include the following:

- simplicity of measurements,
- static character,
- relatively small amount of metal necessary for experimental measurements of ST;
- ability to choose different combinations of cups and substrates;
- the ability to conduct an experiment both in vacuum and in various environments;

The disadvantages of this method are:

- difficulties associated with the need to refresh the surface of the drop;
- difficulty of droplet alignment;
- problems of homogenization of the investigated alloys.

These disadvantages can be minimized by:

- preliminary thermal vacuum treatment of the measuring cell and droplet at higher temperatures than the upper limit of the temperature range of measurements;
- minimizing ellipticity, i.e. ensuring a strictly circular shape of the edge of a special cup for the formation of a liquid drop;
- preparing cups of optimal sizes, taking into account the dependence of the error in photographing drops on its size.

It is known that the error in measuring the ST depends on the size of the drop (the error increases with the size of the drop), and, therefore, there are optimal sizes at which the error in measuring the ST is minimal.

Thus, the large drop method, with the correct consideration of all factors affecting the measurement errors of surface tension, density, and contact angle, can be successfully used for the experimental study of the surface properties and density of metals and their alloys.
As noted earlier, a large contribution to the automation and improvement of the accuracy of static methods for determining the surface tension coefficient was made by Director L.B. with colleagues [9, 10]. In particular, they improved the lying drop method.

The droplet was measured using a high-speed software package that allows processing a digital image of a droplet and optimizing the procedure for determining the ST of a liquid by the methods of a lying drop (on a substrate), “large” drop (in a cup), droplet detachment (hanging drop) [9, 10]. The methodology is based on the ideology of numerical integration of the Young-Laplace equation.

The software complex, in which three blocks are sequentially implemented, was developed as an application for Windows 98 and Windows XP operating systems using the DELPHI programming environment [14].

The image processing unit reads graphic files containing images of the meridional section of the droplet and extracts the droplet profile. The droplet boundary is determined in two stages: 1) digital image processing using a high-pass filter; 2) determination of the contour of the drop by the method of spatial differentiation. The result of the second stage is the dependence of the horizontal section diameter on the drop height.

The computing unit calculates the thermophysical properties of the fluid using linear models. It is possible to use more complex nonlinear models, but in this case, the computation time increases significantly.

The measurement error is determined using the functional representing the sum of the squares of the deviations of the experimental points from the calculated curve:

\[
L = \sum_{i=1}^{N} (y_i - y_i^*)^2,
\]

where \(y_i\) are the coordinates of the experimental points, \(y_i^*\) are the coordinates of the calculated points.

The error in the calculation of the surface tension coefficient is below 2 %, if \(\frac{R_e}{a} < 0.05\), where

\[
a = \sqrt{\frac{2\sigma}{\rho g}}
\]

is the capillary constant, \(g\) is the acceleration of gravity.

The results output unit generates a report file for processing experimental data.

The second group of tasks is devoted to the study of the contact angle \(\theta\). When developing this problem, a whole range of different methods are used [15, 16]. Quite accurate values of \(\theta\) can be obtained using the lying drop method. In this method, it is important to take into account the critical value of the volume \(V_c\) of the liquid, after reaching which, the increase in the height of the droplet should stop, while at volumes \(V > V_c\), the extreme height of the droplet \(h_0\) remains constant [17]. Another problem is the study with digital cameras of the three-phase contact zone with incomplete wetting of the substrate surface by a drop of melt. In particular, due to the high vapor pressure near the three-phase contact, some errors in the estimation of the drop contour are possible.

But still, the determination of the contact angle and other parameters from the digitized images is the most effective and often implemented in practice method, leaving at the same time a large number of unsolved problems associated with its application, especially at the stage of analyzing the digitized field.

Therefore, the main goal at the stage of image processing is to level the profile distortions generated by heat flows and to minimize the error in determining the design parameters of the system. This result can be achieved by successively solving the following tasks: determining the boundary points of an object based on gradient methods; the establishment of deformed sections of the profile; interpolation of the values obtained at the previous stage; finding the coordinates of the profile and the required physical parameters.

The main goal of the first stage of the implementation of the proposed algorithm is to determine the boundary points and localize the deformed sections of the profile.

It is known that the process of droplet formation in some cases is accompanied by the formation of a precursor film or simply a precursor, i.e. film spreading in front of a moving liquid meniscus. In
particular, for various kinds of melts, spreading at the microscopic level is indeed accompanied by the outflow of liquid monolayers in front of the three-phase contact line.

Recently, a new software approach has also been used to determine the contact angle from the parameters of a photograph of a drop. This approach is implemented using the ImageJ application [18] freely distributed by the Swiss Federal Institute of Technology (link: bigwww.epfl.ch/demo/dropanalysis). This application is one of the most powerful tools for processing any images obtained in various physical, chemical and technological experiments. It contains many options for image filtering, edge enhancement, clarity, sharpness, Fourier transform, etc. However, to determine the contact angle, two plug-ins must be loaded into the program: DropSnake and LB-ADSA, respectively for the cases of \( \theta > 90^\circ \) and \( \theta < 90^\circ \).

For successful implementation, the user needs to randomly arrange the nodes on the drop contour and close the profile by clicking on the initial node (this user step is displayed in blue on the screen). Next, you need to click the "snake" window and the application will automatically begin to approximate the drop contour as close as possible to the exact solution of the corresponding drop contour equation (this program step is displayed in red on the screen).

### 3.2 Errors in measuring surface tension by the method of maximum pressure in a droplet

In the method of maximum pressure in a drop, ST is determined by the Cantor formula. Let us consider the validity of those fundamental assumptions and assumptions that were made in the derivation of this formula. When deriving this formula, the following assumptions were made: the drop is strictly spherical, the maximum pressure in the drop does not depend on the wetting angle and the drop radius is equal to the inner radius of the capillary through which the drop is forced. In the case of a liquid-vapor interface being far from the critical temperature, the density of the vapor phase is neglected \( (\rho_1 << \rho_2) \) and the Cantor formula takes the following form:

\[
\sigma = \frac{r}{2} P_m \left[ 1 - \frac{2}{3} \frac{g \rho r}{P_m} - \frac{1}{6} \frac{g^2 \rho^2}{P_m^2} - r^2 \right]
\]

where \( P_m \) is the maximum pressure in the drop, \( r \) is the radius of the capillary, through which the drop is forced, \( g \) is the acceleration of gravity, \( \rho \) is the liquid phase density.

The assumptions made in the derivation of this formula increase, first of all, the requirements for the used capillary [1]. On the one hand, the assumptions about the strict sphericity of the drop and the equality of the radius of the drop to the inner radius of the capillary require that the radius of the capillary be small. However, with a decrease in the radius of the capillary, its ellipticity increases, and, consequently, the relative measurement error of the radius value. Therefore, each metal has its own optimal radius size, and the correct choice and precise determination of the radius is important when making accurate measurements of the ST.

Surface tension is calculated by formula (5), in which, due to smallness, the third term in the Cantor's expression is not taken into account. In this case, an error is allowed, the value of which depends on the radius of the capillary. For \( r << 0.3a \), where \( a \) is the capillary constant, the error less than 0.3\% [1]. The total error in surface tension measurements is the sum of errors in determining the following values:

\[
\frac{S_{\sigma}}{\sigma} = \sqrt{\left(\delta \sigma\right)^2 + \left(\frac{S_{\rho}}{\rho}\right)^2 + \left(\frac{S_{\rho}}{\rho}\right)^2 + \left(\frac{S_{f}}{h}\right)^2}
\]

The value of the height \( h \) is measured multiple times (at least 10 times). In our studies, the coefficient of variation of this value at a confidence level of 0.95 was 0.1\%.

The main contribution to the error in determining the ST by the method of maximum pressure in a drop is made by the error in determining the radius of the capillary through which the capillary is forced out. Manufacturing a capillary with the required diameter that meets stringent requirements is a complex and extremely time-consuming task. Suffice it to say that hundreds of capillaries of different diameters are manufactured to select a capillary of the required diameter. At the same time, stringent
requirements are imposed on the capillary in terms of the presence of internal chips and ellipticity. Capillaries are chosen with a microscope. A preliminary section of each capillary is carefully examined under a microscope and such capillaries are selected, in which the section does not have chips, cracks or other damage. If internal chips or cracks are found, the capillary is rejected. This is due to the fact that until now the degree of influence of internal chips and cracks on the measured value of ST has not been clarified.

The radius of the capillary is determined using a microscope (the accuracy is $10^{-6}$ m.) If the radius of the capillary and its ellipticity are within acceptable limits, then, to determine the average value, multiple measurements of its radius are made. The average radii of the capillaries used in our laboratory to measure the ST of low-melting metals ranged from $2 \cdot 10^{-4}$ m to $3 \cdot 10^{-4}$ m.

When choosing a capillary, it is necessary to take into account an important circumstance associated with the maximum height of the metal column at which a drop breaks off. At large radii of the capillary, this height is insignificant, which decreases the time of droplet formation, and, consequently, decreases the time of measurement. Insufficient time for the formation of a drop can negatively affect the final results, since this violates the conditions of stationarity. An excessive increase in the droplet formation time can also adversely affect the ST measurement results, since this increases the likelihood of uncontrolled external influences (for example, vibration). In our experiments, the height of the metal column varied from 3 to 6 cm, and the measurement time was from 3 to 8 minutes.

Since the capillaries are made of the glass tubes, ellipticity cannot be avoided. Ellipticity introduces an error in the measurement of surface tension, which must be taken into account. A rigorous accounting of this error is difficult due to the absence of a reference liquid with a known value of surface tension. The maximum possible value of this error can be found if we assume that the error in measuring the radius of the capillary is equal to its ellipticity. Obviously, this approach leads to a significant overestimation of the measurement error of the ST, but other, more rigorous methods for assessing this error have not been developed to date. Therefore, when indicating this error, it is necessary to stipulate each time that this is the maximum possible error in measuring ST.

The ellipticity of the capillaries used in our laboratory did not exceed 0.5 %. Therefore, we assume that $S_r = 0.006$, although the real value should be much lower.

Calculations of the maximum total error in measuring the surface tension show that the coefficient of variation of the surface tension at a confidence level of 0.95 is less than 0.8. Then, assuming the value of surface tension on average equal to 500 mJ/m$^2$ for the standard deviation, we have: $S_\sigma = \pm 4$ mJ/m$^2$.

We have calculated the maximum possible error in measuring the surface tension. It should be noted that the scatter of the experimental points for this device is characterized by the last two terms in (6), and this is 0.2 % or $\pm 1$ mJ/m$^2$. When studying the temperature or concentration dependence, it is this error that is of greatest interest.

The gravitational technique of P.P. Pugachevich, having a number of advantages over other methods of measuring ST, has two fundamental important disadvantages. This is, first of all, a narrow temperature range in which measurements can be made ($T_m$ below 773 K), due to the low softening temperature of the glass from which these devices are made. The use of quartz devices slightly increases the upper limit of the temperature range. However, at high temperatures, quartz becomes transparent to inert gases (i.e., allows inert gases to pass through), which cannot but affect the measurement error of the ST. The second important drawback of this method is due to the presence of fundamental difficulties in introducing modern information technologies into the measuring process. However, despite the indicated drawbacks, the results of experimental measurements of ST by this method, subject to all methodological recommendations, are highly accurate.

When analyzing the errors in measuring the ST by this method, one should pay attention to one fundamentally important circumstance, which until now has remained unnoticed. As will be shown below, a critical analysis of the influence of various factors on the ST measurement errors shows that the overwhelming majority of these factors are aimed at lowering the measured ST value. We
examined in detail the mechanism and the degree of influence of each of them on the ST measurement results.

4. Results
To analyze and accurately determine the measurement error of a physical quantity, it is necessary to find the error distribution function. Usually, when solving this problem, it is assumed that the random measurement error is distributed according to the normal law. However, as a rule, there are no serious grounds for such assumptions. In this regard, the experimental verification of the hypothesis about the form of the distribution function of the random error when measuring any physical quantity is of scientific and practical interest.

To determine the distribution function of a physical quantity, a large array of experimental data is required. For these purposes, the author of [1] used the experimental data on the surface tension of low-melting melts obtained in our laboratory.

Since in experimental studies of ST each measurement is repeated, as a rule, several times, for these purposes it is possible to use samples of 8–10 measurements with different values of the mathematical expectation. Since the form of the distribution function does not depend on the value of the mathematical expectation, individual samples can be reduced to a zero value of the mathematical expectation, which makes it possible to collect statistical material sufficient to construct an experimental histogram and a theoretical distribution curve. For instance, for the construction of the histogram, more than 8000 measurements were used [1].

The calculations performed and their comparison with theoretical calculations showed that random errors in measuring the surface tension by the maximum pressure method in a drop are distributed with high accuracy according to the normal law. In this case, the surface tension is also determined with high accuracy (the dispersion is 0.32 mJ/m$^2$).

In such an analysis of the measurement error, it is necessary to take into account that the total error consists of both random and systematic components. By increasing the number of measurements, the random error can be reduced until it becomes negligible. In this case, the value of the total measurement error is determined by the systematic error.

Let us consider in more detail the systematic error in measuring the ST in the method of maximum pressure in a droplet. The systematic error is influenced by the following factors:

- errors in measuring the internal radii of the capillaries;
- the presence of ellipticity and chips on the inner sections of the capillaries;
- errors in reading the height of the meniscus;
- diameter of the manometric cylinder vessel;
- the presence of at least a slight wetting of glass by the melt (partial wetting, as a rule, is observed at low vacuum or insufficient purity of metals or in the presence of oxides);
- optical distortion due to irregularities on the outer walls of the manometric and measuring vessels;
- fixing the height of the metal column ahead of the time required to reach the ultimate pressure;
- vertical installation of the capillary in the measuring devices.

In this regard, let us consider in more detail the influence of each of the listed factors on the error in measuring surface tension and density. For convenience, it is desirable to divide these factors into two groups.

The first group includes factors that cause systematic errors, which by definition take both positive and negative values. Among the errors listed above, these include errors caused by optical distortions of the capillary edge and profile, drops due to irregularities in the walls of glass tubes, errors of measuring instruments in determining the internal radii of capillaries and the height of the metal column above the edge of the capillary.

As noted above, the values of these errors, in addition to the errors due to optical distortions, are determined by the accuracy of the instruments with which these quantities are measured. The inner radius of the capillary and the height of the meniscus are determined, respectively, by an optical
microscope and a cathetometer with a sufficiently high accuracy. With repeated repetition of measurements, the random component of the measurement error of these quantities can be minimized, and the systematic error can either increase (be positive) or decrease (be negative) the measured value of the ST.

A fundamentally different situation arises when analyzing the influence of other factors on the measured value of ST. The difference between these factors, which we conditionally included in the second group, is that they do not increase, but always lower the ST value. We attributed the following factors to the second group:

- the presence of ellipticity and chips on the inner sections of the capillaries;
- the effect of the sphericity of the liquid surface in a manometric cylinder;
- the presence of at least a slight wetting of the glass by the melt (partial wetting is observed at low vacuum, insufficient purity of metals or in the presence of oxides);
- fixing the height of the metal column ahead of the time required to reach the ultimate pressure;
- the influence of various factors, in particular vibration, on the stability of a drop of metal, which, when the required pressure is reached, “bursts”, i.e. breaks off the capillary cut;
- deviation of the measuring capillary from verticality during measurements.

5. Discussion and conclusions
The stated factors have an uncontrollable effect on the final results and lead to errors in the measurement of ST. The question arises as to which group these errors belong to random or systematic ones. If they were random, then by increasing the number of measurements the experimenter could reduce them to a minimum. However, it is not. If we classify them as systematic, then problems arise due to the fact that they never increase, but always lower the measured value of ST. Consequently, these errors have their own specifics and when analyzing the total error in measuring the ST, they must be considered taking into account these features.

In conclusion, it should be noted again that these factors, without individually having a noticeable effect on the final results, collectively (in total) lead to a noticeable decrease in the measured value of ST.

This, in our opinion, is due to the fact that the method of maximum pressure in a drop always gives somewhat lowered ST values for all low-melting metals.

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