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Density of tin, silver and copper

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ABSTRACT

Purpose: Purpose of this paper is to report on the development of a new density measurement cell.

Design/methodology/approach: Measurement cell based on Archimedean principle and consisting of induction furnace and a high/precision balance was applied for measurement of tin, silver and copper density.

Findings: It was found that new cell is suitable for high temperature measurement of liquid metals density at temperatures from 700 to 1520°C. Measurement results are in a good agreement with the literature values. Density deviates by 0.5-1% depending on the metal.

Research limitations/implications: Accuracy of the density measurement decreases at temperatures below 700°C due to oxidation of the melt surface. More accurate data on thermal expansion coefficient for sinker material is required.

Practical implications: Experiments showed applicability of the new measurement cell. Archimedean principle is among the most sensitive density measurement techniques. New cell will be further used for measurement of iron-based alloys. Problems of measurements are discussed.

Originality/value: Paper describes application of the known density measurement technique. The paper is of interest for the material scientists working with high-temperature thermophysical properties measurements and users of thermophysical properties data.

Keywords: Density, Tin, Silver, Copper, Archimedes

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

Thermophysical properties of liquid metals play an important role in theory and process metallurgy. Data on viscosity, surface tension, density of metal and alloys are inevitable for modelling, design and control of metallurgical processes where the liquid state of metals takes place. It is also known that cast product quality correlates with liquid characteristics of the melt [1]. But, when it comes to the real practice of experimental investigations of liquid phase properties, researches usually face significant challenges in proper measurements of density, surface tension and viscosity. Most of the liquid metals are high-temperature systems and often chemically aggressive. Such nature of liquid metals poses strict requirements to measurement equipment, which should: 1) resist high temperatures (800-2000°C depending on metal); 2) does not/minimally react with molten system; 3) provide nonreactive atmosphere around the melt to protect from oxidation. These problems are especially actual for the measurements of liquid iron-based alloys.

Among the density measurement techniques, the Archimedes' principle is recognized as one of the most precise ones [2]. There are direct and indirect method. Direct one is based on immersion of the body with the known volume into the studied liquid. The weight change (buoyancy force) of the immersed body is equal to the weight of displaced liquid. Present work reports results of development of a new density measurement cell based on Archimedes' principle in Institute of Iron and Steel Technologies of TU Bergakademie Freiberg, and its application to density estimations of 3 reference materials: tin, silver and copper.

2. Research methodology

In a present research the density of the 3 technically pure metals was investigated with the use of the newly developed measurement cell based on Archimedes' principle. Equipment has been created with the purpose to measure density of molten steels and iron alloys.

2.1. Materials

Three different metal samples were used for experiments: tin sample supplied by Merck (99.99% purity); silver sample supplied by Merck (99.99% purity); and electrolytic copper sample with a purity of 99.9%. Copper samples were preliminary cleaned with the sandblasting to remove the surface oxides, while the two other metals were ultrasound-cleaned only with ethanol.

2.2. Measurement equipment

Samples were molten in a laboratory induction furnace (Fig. 1). The furnace can be moved in 3 dimensions. Vertical position of the furnace is controlled with the micrometre precision. The density was measured by immersion of sinkers' assembly, hanged to the bottom connection of the balance Mettler-Toledo XS105. The balance has an accuracy of 0.02 mg. In present research 2 ceramic sinkers (ZrO_2 stabilized with MgO) of 0.346 and 1.639 cm^3 were used as immersion bodies. Volume of the sinkers was estimated by immersion of them into the distillate water at room temperature. Thermal expansion coefficient of the ZrO_2 is assumed as $10.1*10^{-6}$/°C (as given by supplier).

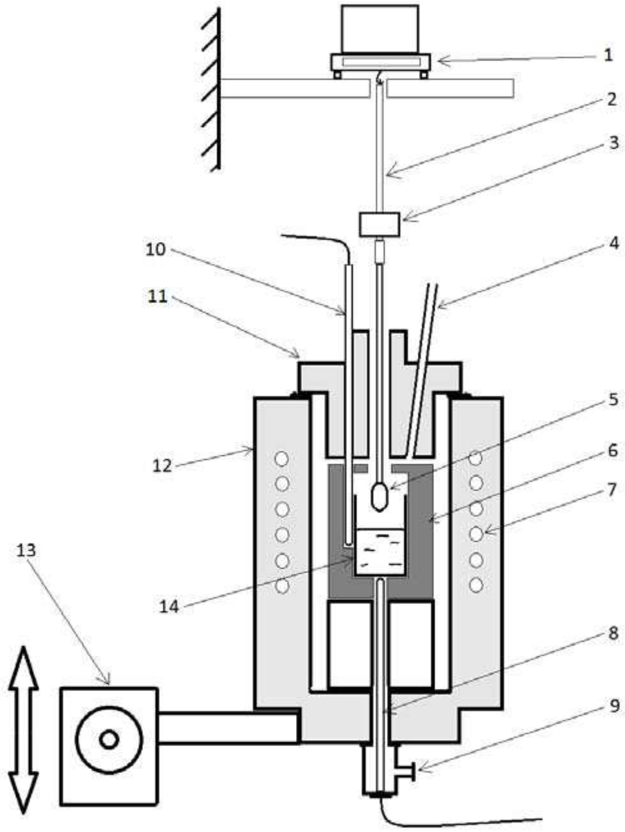

Fig. 1. Scheme of the laboratory measurement cell for the determination of density with the use of Archimedes' principle. 1 – Mettler-Toledo XS105 balance placed on a fixed platform; 2 – molybdenum rod; 3 – extra load (Cu); 4 – top input of gas (argon); 5 – ceramic sinker; 6 – graphite heating crucible; 7 – inductor coil; 8 – bottom thermocouple (type B); 9 – bottom gas input (argon); 10 – side thermocouple (type B); 11 – ceramic cover of the reaction chamber; 12 – ceramic furnace; 13 – electric drive for vertical positioning; 14 – graphite crucible

In order to prevent oxidation of the samples, they were molten in ZrO_2 crucibles placed inside graphite heating body. The inner volume of the furnace was flushed with argon (250 l/min). Argon purity was measured as <5 ppm of O_2 on the entrance of furnace. At the temperatures above 700°C the inner volume of the furnace showed no detectable oxygen due to the Boudouard reaction of any incoming O_2 with the graphite heating body. Such a simple solution helps to eliminate the undesirable oxidation of the molten samples (although it does not work for low melting point metals such as tin or lead).

Temperature in the melt was controlled in indirect way. Two thermocouples of type B (Pt/Pt-Rh) were used: one attached to the bottom of the crucible and another one attached to the outer side of the crucible on the level of 20 mm above the bottom. Calibrations showed that temperature in the melt was 10 ± 2°C lower than in a side thermocouple.

2.3. Measurement procedure

Samples were heated with the rate of 25°C per minute. The holding time was > 30 minutes before each data point measurements. Each temperature point was investigated by subsequent immersion of two sinkers into the molten sample. The sinkers were immersed on the target level 3 times for 5 minutes at every temperature. The average value that was recorded by the balance in a last minute of 5-minutes interval was considered as a weight change of the sinker. The density was calculated as a weight change difference between two sinkers divided by volume difference between two sinkers. This way the effect of surface tension on the sinker holder was cancelled.

3. Description of achieved results of own researches

Density measurement results for 3 studied metals in comparison with the recommended values from literature [3,4] are present in Figure 2 and Figure 3. As can be seen from Figure 2 and Figure 4, at elevated temperatures measurement cell shows almost constant overestimation of the density on 1% for Ag and 0.5% for Cu (on average). Cu has about 30% higher surface tension than Ag [5] but slightly lower deviation from the recommended density. So, the data deviation cannot be clearly explained by the effect of surface tension.

Analysis of the density data for tin should consider the specific feature of the measurement cell. First, the reactor zone is not fully gas-tight. Furnace chamber provides protection of the tin sample from oxidation only starting from approximately 700°C (when both the Boudouard reaction starts and SnO_2 reduction becomes possible). This feature leads to uncontrolled formation of oxides on the surface of the melt. Probably, the oxides on the surface are the reason of large at low temperatures and decreasing at higher temperatures deviation for the density of tin (Figs. 4 and 5).

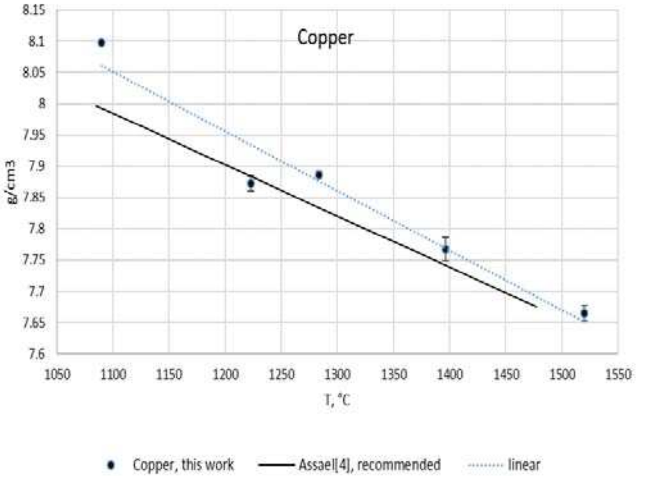

Fig. 2. Density of copper measured in a range of 1090-1520°C compared to recommended values [4]

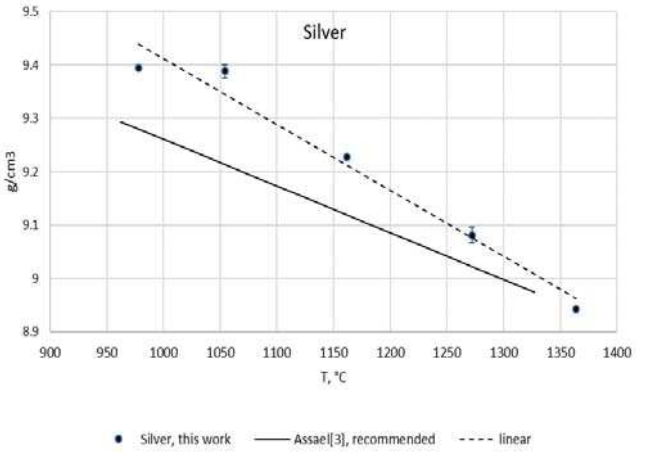

Fig. 3. Density of silver measured in a range of 978-1364°C compared to recommended values [3]

During the experiments it was important to check the deviation of weight change in multiple immersions at same temperature. It helped to optimize the measurement procedure towards less immersions number. Analysis of the standard deviations of weight change at same temperature revealed very low difference – maximum was 2.3% (Tab. 1). It means that in future experiments number of immersions can be limited to only one immersion. In such procedure the number of temperature points that can be measured during the reasonable time (6-8 hours) will significantly increase.

Table 1.
Equations describing the density lines of silver, copper and tin

Metal	Equation, °C	Correlation coefficient R^2	Max standard deviation of three immersions, %
Tin	$\rho = 7.3926 - 11.26*10^{-4}T$	0.863	2.312
Silver	$\rho = 10.6417 - 12.34*10^{-4}T$	0.9708	1.547
Copper	$\rho = 9.1023 - 9.581*10^{-4}T$	0.9476	1.877

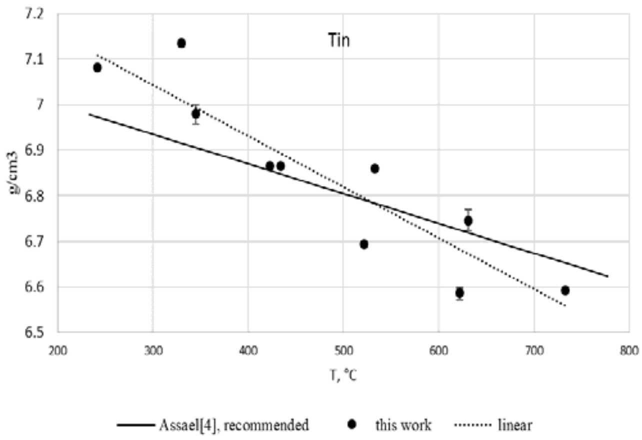

Fig. 4. Results of 2 measurement of tin density in a range of 242-733°C compared to recommended values from literature [4]

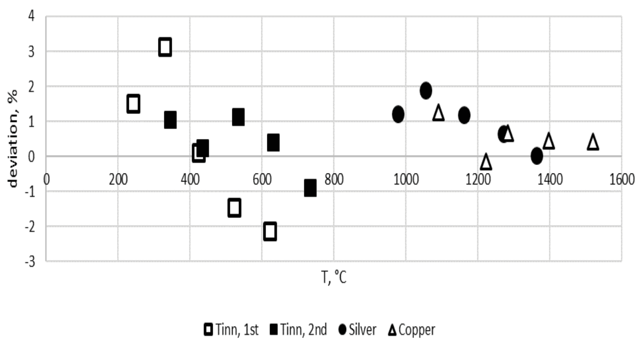

Fig. 5. Deviation from the recommended values

4. Conclusions

Calibration of the newly developed density measurement cell was successfully accomplished. With the use of Archimedes' principle, density of three liquid pure metals was measured. The following results has been obtained:

- Density of noble metals was measured with overestimation of approximately 1% for silver and 0.5% for copper. This value can be further used as a correction coefficient for the measurement of liquid steels density.
- Measurement cell proved to deliver relatively precise data at temperatures up to 1520°C.
- At the same time, measurement cell showed limited ability to deliver precise data at temperatures below 700°C due to absence of full protection from oxygen inflow into the furnace.

The new equipment has a high potential to be used for measurements of liquid steels density.

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Additional information

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