

Application Report

Contact angle measurement at high temperatures

Application report: AR238e
Industry section: Metallurgy
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Date: 12/2003

Method:



Keywords: High temperature, slag melts, contact angle



Drop Shape Analysis System
DSA10HT

Contact angle measurement at high temperatures

Measuring the contact angle of slag melts on graphite and aluminum oxide carrier materials

Abstract

The contact angle of molten slag has been determined on various carrier materials (graphite and Al_2O_3) under a protective gas atmosphere. An optical contact angle measurement based on the sessile drop method using the Drop Shape Analysis System DSA10HT and a high-temperature tube furnace was used as the quickest and simplest method for measuring the wetting.



Fig. 1 shows the Drop Shape Analysis System DSA10HT.

Introduction

Measuring apparatus

A well-proven combination of optical drop shape analysis (which can be carried out fully automatically by the built-in control software) and an innovative tube furnace that can be used up to 1900°C was used for making the contact angle measurements.

The optical drop shape analysis system functions on the basis of a video-supported digital image recording and evaluation technique. By using the method of a sessile drop on a solid surface and a fit method adapted to suit this rotationally symmetrical profile (e.g. Laplace, ellipse, conic section, circle fit) it is possible to use a numerical and analytical drop profile evaluation method both online and offline to determine the contact angle. Images can be stored as snapshots or as movie sequences by the movie function included in the software.

In order to make contact angle measurements on the molten slag a horizontally positioned tube furnace was used; an inert gas atmosphere was used for these measurements. In principle this furnace can also be used under oxidizing or reducing atmospheres up to 1900°C. If an appropriate insert tube is used the furnace can also be operated under vacuum. The furnace can be operated in both a horizontal and vertical position. If necessary it can also be fitted with a water-cooled jacket.

The heart of the furnace is the working tube. It consists of a dense, sintered high-purity ceramic material. The working tube openings are sealed with ISO-KF standard flanges used in vacuum techniques, Swagelok connectors are used for the gas supply. The heating element is located directly on the working tube. The whole heating chamber is filled with inert gas to avoid oxidation processes. Atmospheric air is excluded either by a weak flow of inert gas or the housing is hermetically sealed.

The working tube is located in the heating chamber. Air contact is only possible via the flange openings. There is no connection to the heating chamber atmosphere. This means that work can be carried out in the working tube under any atmosphere, which can differ from both the surrounding atmosphere and that in the heating chamber. Work can also be carried out under vacuum up to the temperature at which the working tube loses its mechanical stability. If the composition of the sample material is known then the flow of inert gas can be led through the working tube and then through the heating chamber. As the hot parts of the working tube are surrounded by inert gas, this means that even at the highest temperatures no oxygen can diffuse into the working tube. As a result the furnace is particularly suitable for heat treatment under the exclusion of oxygen. A special feature of this furnace is the sample positioning by using a mobile sample stage; this can be seen in the view of the sample chamber shown in Figure 2.

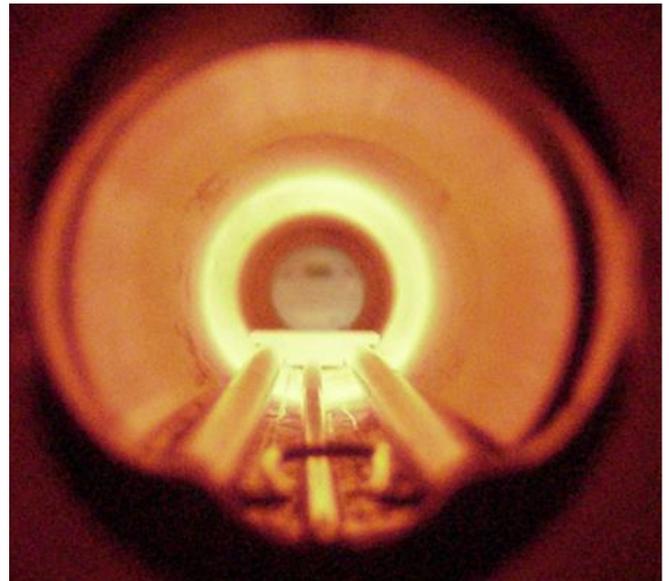


Fig. 2: Sample holder in interior of furnace

Measuring parameters

Contact angle measurements were made in the temperature range $T = 1300^{\circ}\text{C} - 1750^{\circ}\text{C}$. The measuring parameters used were as follows:

Contact angle type: Static contact angle

Fit method used:

Ellipse fit for slag on aluminum oxide

Laplace fit for slag on graphite

Measured values/Results

Contact angle measurements between slag and graphite were made in the temperature range $1400^{\circ}\text{C} - 1750^{\circ}\text{C}$ under protective gas.

The course of the melting process can be seen from the examples shown in Figures 3 to 7; these show the initial condition of the sample before melting, the melting process itself and the stable drop at the end of the melting process.

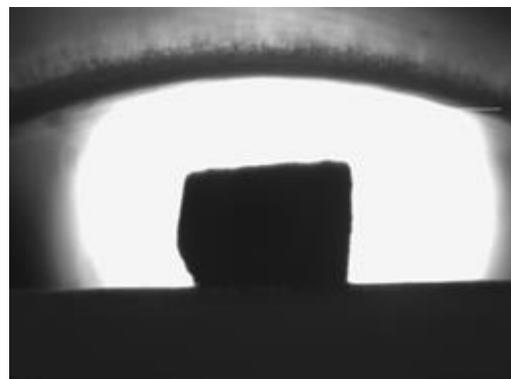


Fig. 3: Slag sample on graphite at approx. 1160°C

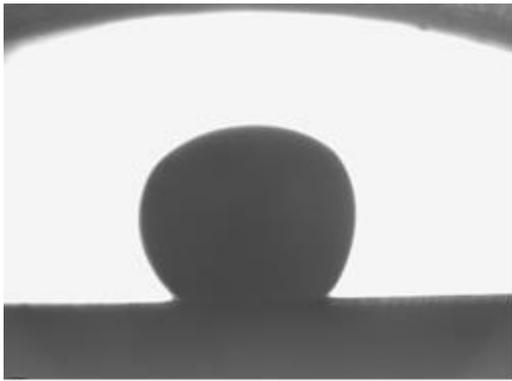


Fig. 4: Slag sample on graphite at approx. 1420°C



Fig. 5: Slag sample on graphite at approx. 1635°C

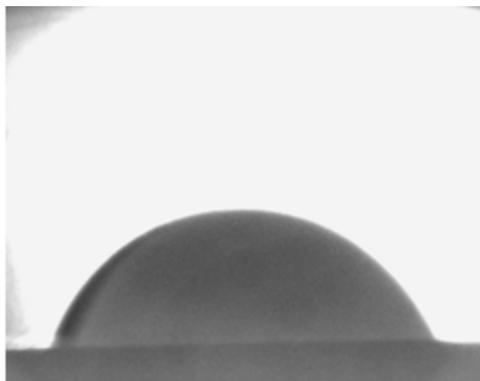


Fig. 6: Molten slag on graphite at approx. 1685 °C



Fig. 7: Molten slag on graphite at approx. 1700°C

The sequence of images documented in the temperature range 1635 - 1700°C can be evaluated with respect to the following functions:

- Contact angle as a function of the temperature
- Contact angle as a function of the drop age
- Drop diameter as a function of the drop age
- Drop volume as a function of the drop age
- Contact angle as a function of the drop volume
- Contact angle as a function of the drop diameter
- Drop volume as a function of drop diameter.

To demonstrate one of these possibilities we have chosen the function "Contact angle as a function of the drop age of the melt"; this is shown in Fig. 8.

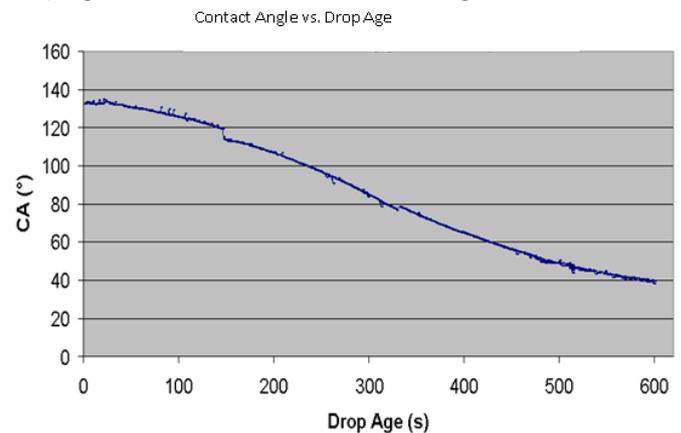


Fig. 8: Contact angle of slag on graphite as a function of the drop age of the melt

Contact angle measurements between slag and Al_2O_3 were made in the temperature range 1310°C – 1510°C. Drop images were evaluated in this temperature range, a selection of these are shown below (Fig. 9-14).

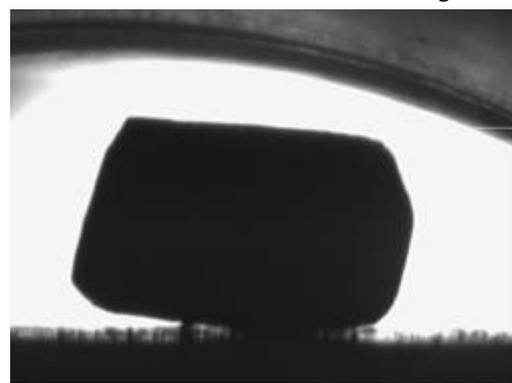


Fig. 9: Slag on Al_2O_3 at approx. 1100°C



Fig. 10: Slag on Al₂O₃ at approx. 1365°C

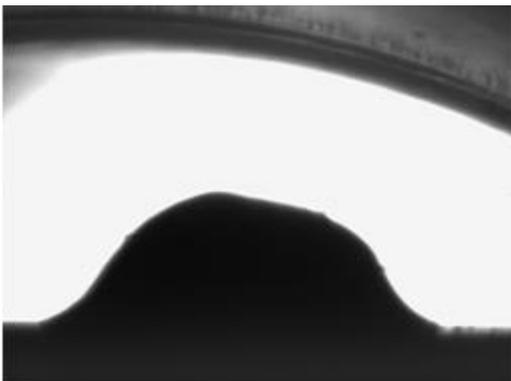


Fig. 11: Slag on Al₂O₃ at approx. 1410°C

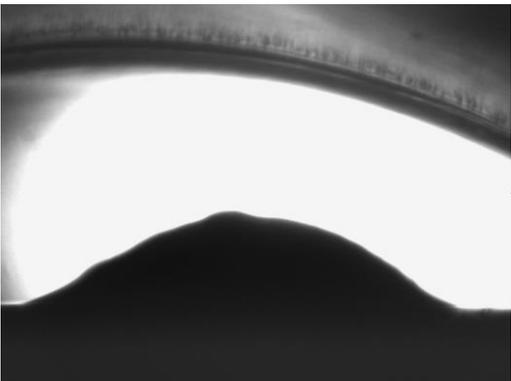


Fig. 12: Slag on Al₂O₃ at approx. 1435°C

The slag on the Al₂O₃ carrier melted at a considerably lower temperature than the slag on graphite and without the formation of a spherically shaped sample.

By making an appropriate software implementation it is still possible to evaluate the contact angle on such non-spherical melts.

Summary

As a result of our contact angle measurements in the range $T = 1300 - 1750^\circ\text{C}$ we would like to briefly summarize the advantages of the experimental system described above:

The tube furnace used does not have any exposed ceramic. A getter was used with the tube furnace to ensure an oxygen-free working atmosphere, so that with the appropriate flow of inert gas no oxygen diffusion into the working tube from outside was possible. If the composition of the sample is known then the flow of inert gas can be led through the working tube and then through the heating chamber.

The choice of the heating element in combination with the appropriate working tube allows working temperatures up to 1900°C.

On request the furnace can be equipped with a mobile sample holder (with built-in thermocouple).

The choice of the ceramic carrier material required for the melting process depends on the material to be melted and the temperature range required for the melting process.

If an appropriate tube is used then the tube furnace can also be used under vacuum. Evacuation before the start of the measurement allows absolute freedom from oxygen to be guaranteed in the sample chamber right from the start of the measurement without large amounts of inert gas being used for purging.

A reference unit DSA10HT for drop shape analysis at temperatures up to 1900°C is available at EHK institute of Montanuniversity Leoben (Austria).