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Solar experimental methods for observing melting plateaux and associated temperature measurements for refractory oxides from 2000 to 3000 °C

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Abstract

Melting points of refractory oxides at high temperature can be obtained through different techniques (resistive furnaces, induction furnaces, laser beams, imaging furnaces . . .). Here, we present a solar furnace experimental setup and methods developed at the Odeillo Solar Furnace (IMP-CNRS) and used to measure melting and freezing plateau temperatures between 2000 and 3000 °C.

We detail a new procedure we have developed that produces a controllable, long duration temperature plateau, and we discuss the appropriate temperature measurement method.

The principle is to heat a self-crucible product with the solar concentrated flux and to preserve the thermal equilibrium at the melting point temperature by providing a continuously regulated solar flux supply. Two critical points are treated: the control of the right level of temperature and its measurement, which is hampered by the solar reflection.

Results obtained with Al_2O_3 (melting point = 2053 °C) are presented as example.

Keywords: Melting point; Al_2O_3; Thermal properties; Solar Furnace

1. Introduction

The first determinations of the melting points for refractory oxides were published in 1910[1] and 1930[2] and, after an interruption, further work was conducted from 1950.[3] To obtain the melting of the oxides, the means of heating have evolved with technology: from oxyacetylene torches, resistive furnaces, induction furnaces and imaging furnaces to power laser beams. The use of a concentrating solar furnace as radiative heat source appeared in 1960.[4]

The present work belongs to this last field. In parallel with the researches devoted to metal–carbon eutectic fixed points in several National Metrology Institutes,[5] we have evaluated the application of a solar furnace to study the suitability of metal oxide phase changes to be used as secondary reference points,[6] on the International Temperature Scale (ITS-90), in the range 2000–3000 °C.

Preliminary results[7] are promising; they incorporate contamination-free melting and the production of the desired freezing phenomena. They are not yet optimal however, and we wish to increase the duration of the freezing plateau and to improve the temperature measurement method. The goal of the work presented here was to improve these two points.

In the first part of this paper, we describe the experimental installation and we detail the observation technique for the initial melting point observation. In the second part, we present the new method and analysis of the optical temperature measurement in the presence of parasitic reflected solar flux. In both cases, we chose alumina to illustrate the experimental results.

2. Experimental set-up and previous method

The experimental set-up and the method for freezing plateau method observation considered here has been devel-

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oped at our Institute for related applications.[4] Both have been recently improved for the purpose of producing a secondary reference point.[7]

To avoid contamination, possible alternatives would be to use the self-crucible technique with a radiative heat source such as a concentrating solar furnace, an imaging arc furnace, or a laser, to reach temperatures above 2000 °C. The radiative heat flux must be around 10 MW/m^2 to melt the material under investigation.

2.1. Solar furnace facility

As illustrated in Fig. 1, our solar facility consists primarily of a 2 m diameter fixed, horizontal axis parabolic mirror (3) with a focal length of 0.85 m. The mirror has an aperture of about 0.2 m in diameter at its center, resulting in a 9°-shadowed cone on its axis. The maximum possible incidence angle is 60°.

The solar beam is reflected to the parabola by a 3 m × 3 m heliostat (6). The incident flux on the parabola can be regulated by means of a primary shutter (8) in the path of the parallel solar beam (9), between the heliostat and the concentrator.

With this facility, an average flux of about 14 MW/m^2 can be obtained over a circle of 0.01 m in diameter, which corresponds to a concentration ratio, C_p, of 14,000.

The experimental device, in this case a rotating cylindrical reactor (1) detailed in Fig. 2, is located at the focus of the concentrator.

The concentrated solar flux (2) can be rapidly and completely switched off by a small water-cooled shutter (5) located on a fast-moving mechanism. This movable shutter, which we will refer to as a moving mask, is designed to eliminate the flux in less than 0.1 s. Moreover, it is large enough to cut all the concentrated flux, and features a central hole corresponding to the non-irradiated solid angle of the 0.2 m parabola aperture. These devices allow measurement without solar radiation, which can have a hampering effect.

The optical pyrometers are located behind the parabola (4) and are focused at normal incidence toward the sample. The measurement distance is about 1 m.

The concentrated solar flux is sufficient to melt the refractory oxides easily; generally times of less than 1 min are required. Steady state conditions are reached after only 1 or 2 min; this should be noted as an important asset of the method The primary shutter and moving mask allow the freezing process to be rapidly started and precisely controlled.

2.2. Melting reactor and formation of self-crucible

The cell containing the material is a water-cooled rotating reactor with a cylindrical geometry (diameter $D = 0.03$ m,

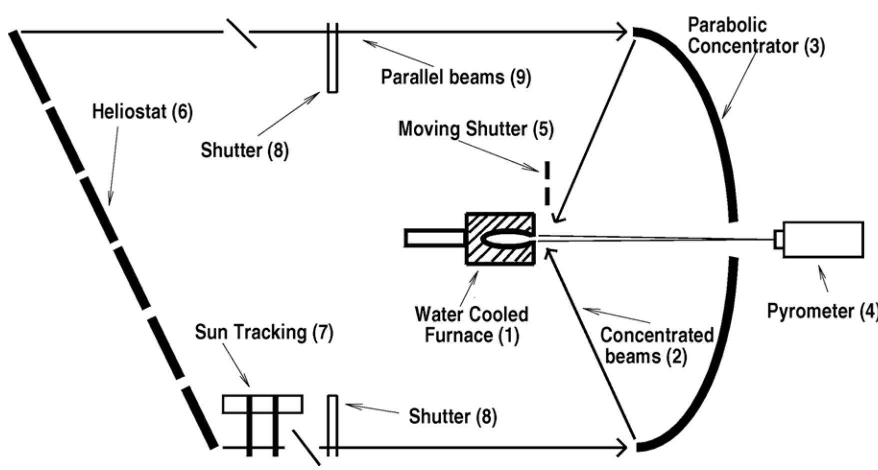

Fig. 1. Schematic diagram of the experimental set-up.

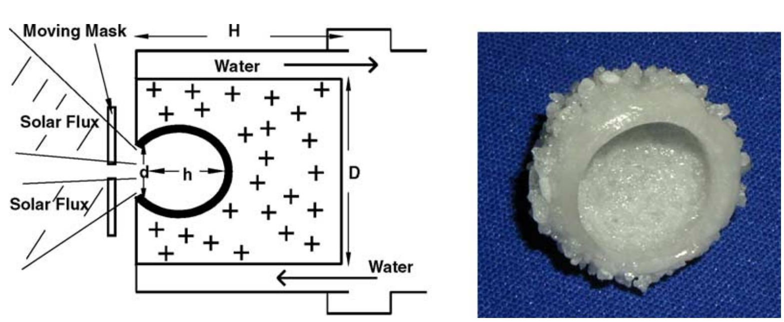

Fig. 2. Rotating reactor for alumina and photo of a cavity.