

PSI's 1 kW imaging furnace—A tool for high-temperature chemical reactivity studies

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Abstract

A new experiment has been installed to conduct studies at temperatures as high as 2500 K on chemical reactions that involve solids or melts and the release of condensable gases. The sample is radiatively heated by a 1 kW xenon short arc lamp placed in the upper focus of a vertically oriented ellipsoid of revolution. The optimal optical configuration has been determined by a Monte-Carlo Ray tracing method. Several methods to machine the reflector have been evaluated by experimentally determining the optical quality of the surface of plane test pieces. In the imaging furnace the sample is placed on a water-cooled support and heated by the concentrated radiation. This arrangement allows for fast heating and impedes the reaction of the sample with crucible material. A remotely controlled hammer allows for freezing the high-temperature composition of the sample by a fast quench. Thus, the sample can be later analyzed by conventional methods such as XRD or TEM. To allow for measurements under defined atmospheres and to protect the ellipsoidal reflector from liberated condensable products, the entire sample stage is enclosed by a hemispherical glass dome. The dome itself is protected from condensable compounds by a laminar flow of inert gas. Experiments with an incense cone at the place of the sample to visualize the gas flow showed that a steady layer of inert gas protects the dome from smoke, if the inert gas flow is properly adjusted. Measured peak flux densities clearly exceed 500 W cm^{-2} required to access temperatures of at least 2500 K. Decomposition experiments on copper sulfides confirmed the operation of the furnace. In the near future flash assisted multi-wavelength pyrometry (FAMP) will be implemented to measure sample temperatures online. Though the imaging furnace was developed to study the decomposition of metal sulfides it is obviously suited to conduct high-temperature studies on most materials relevant for high-temperature solar technology.

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1. Introduction

We recently proposed the extraction of copper from copper sulfide concentrate in a solar furnace

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(Winkel et al., 2003, 2004). The reaction is expected to proceed spontaneously at temperatures above 2000 K under an inert atmosphere. Under these conditions thermodynamic and kinetic data, as they are required for the assessment of the proposed process and for a later design of an efficient solar chemical reactor, are not available in the literature. Thus a new tool had to be fabricated to determine the kinetics of the decomposition reaction and the purity of the product. A conventional furnace or a thermobalance is not suited for this task. Firstly, because temperatures up to 2500 K are required and secondly, because the copper iron sulfides melt under these conditions and contamination of the samples by the crucible is likely. The formation of melts and condensable gases as well as the high working temperatures prompted the development of an imaging furnace. Its design is in part inspired by the solar chemical reactor TREMPER (Frey et al., 2001), which was used to obtain kinetic and thermodynamic information on the reduction of metal oxides at temperatures between 2000 and 2200 K under well-defined atmospheres (Frey et al., 2000). In TREMPER, samples are irradiated with concentrated solar radiation while resting on a water-cooled metallic sample holder. Most melts do not wet the cold metal but form a small droplet which minimizes the contact area and thus the reaction between sample and sample support. The inhomogeneous irradiation introduces strong convective mixing, as shown by the fast moving thin layer of slag on the surface, thereby hindering segregation phenomena as evidenced by the visual inspection and XRD analysis of the sample. The same principle is employed for the sample holder of the imaging furnace, where the sun is replaced by a 1 kW arc lamp as radiation source. In the following, we report on the design process of the imaging furnace and first results of its characterization.

2. Experimental

The Monte-Carlo based ray tracing program VeGaS (Petrasch, 2002) was used to evaluate the optimal configuration of the furnace and to predict its performance. VeGaS performs at about 2000 rays/s on a 500 MHz Pentium III computer. The semi-axes a and b of the ellipsoidal reflector and the reflectivity R were kept fixed ($a = 480$ mm, $b = 420$ mm; $R = 0.9$). The 1 kW xenon short arc lamp (OSRAM XBO-1000W HS/OFR) was approximated by a Lambertian sphere with a diam-

eter of 2 mm. Shading due to the electrodes was taken into account by choosing the appropriate angular probability distribution (Itchner, 2002). The sample was approximated by a half-sphere with a diameter of 5 mm, with its center coinciding with the second focus of the reflector.

The surface quality of the aluminum reflector produced by different machining techniques was qualitatively analyzed by comparing a beam of white light reflected by plane sample surfaces at an angle of 40° recorded with a Nikon 750 CoolPix digital camera. For a quantitative assessment the beam of a He-Ne Laser was reflected off the test surface and imaged with an ICCD-camera (La Vision, DynaMight 5/18-02, photocathode S25) as it hits a plane Lambertian target with a known reflectivity. Light levels were adjusted by neutral density filters (Andover). The flux density distribution on the water-cooled reference target (copper, powder-coated with $\text{Al}_2\text{O}_3\text{-TiO}_2$ (97–3 wt.%)) in the sample plane of the imaging furnace was measured by an absolutely calibrated ICCD camera (Andor, Type-Nr. DH510-18F-01, with a A-CC010 controller; gain: 1; gate: 1000 ns; Nikkor 105 mm lens).

3. Results and discussion

3.1. Evaluation of the components

The symmetry of the lamp and of the reflector suggest four possible orientations of the arc lamp relative to the ellipsoidal reflector (see Fig. 1). For each configuration the fraction of the radiation finally reaching the sample and the uniformity of

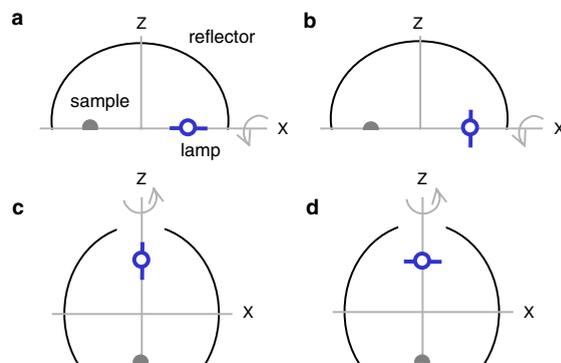


Fig. 1. Horizontal (top) and vertical (bottom) orientation of the reflector with the lamp parallel (left) or perpendicular (right) with regard to the axis of revolution.

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