

Using confocal scanning laser microscopy for the in situ study of high-temperature behaviour of complex ceramic materials

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Received 14 October 2006; accepted 20 January 2007

Available online 30 April 2007

Abstract

The confocal scanning laser microscopy (CSLM) technique has been successful in many metallurgical fields. This paper assesses its applicability to the in situ investigation of the high-temperature behaviour of complex ceramic materials. Magnesia–chromite refractory is selected as a ceramic test material. At room temperature, CSLM images correspond well to typical light optical microscopy (LOM) and backscattered electron (BSE) micrographs. In fact, because of the high axial resolution (short focal depth) and the confocal optics of the CSLM technique, the porosity level of the mirror polished ceramic specimens is more truthfully assessed by the CSLM (2-D) images than by the BSE micrographs (long focal depth). However, at high-temperatures (1550–1650 °C) the observed CSLM (2-D) image quality is slightly poorer. The principal explanation is the in situ roughening of the specimen surface during heating. The roughening has two causes: differential thermal expansion of the two primary phases in the ceramic test material and, to a lesser extent, thermal grooving. Nevertheless, it is shown that the CSLM image quality suffices for an in situ study of the high-temperature behaviour of ceramic materials. This is illustrated for the magnesia–chromite system by examining the dissolution mechanism of secondary (magnesiochromite) spinel into the periclase phase.

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Keywords: Electron microscopy; Thermal expansion; Spinel; Refractories; Confocal scanning laser microscopy

1. Introduction

By combining the advantages of a laser, confocal optics and an infrared image furnace (IIF), confocal scanning laser microscopy (CSLM) is a powerful technique that enables high-temperature in situ observation of a number of metallurgical phenomena.

Since the pioneering work of Emi and co-workers on real-time investigation of crystal growth and transformations in Fe–C alloys,^{1,2} a multitude of successful studies have been performed. This includes the in situ observation of solidification and high-temperature phase transformations of steel and iron alloys,^{3–5} the inclusion behaviour on liquid steel^{6,7} and stainless steel⁸ surfaces, the inclusion behaviour during steel solidification,^{9–12} the inclusion behaviour at molten steel/slag interfaces,^{13,14} the Marangoni flow at the solid/melt interface of steel,¹⁵ the dissolu-

tion of oxidic inclusions (or microparticles) in slags transparent to laser light,^{16–22} and the crystallisation behaviour of slags.^{23,24}

In the present work, it is assessed if the CSLM-IIF technique can also be applied to the investigation of the high-temperature behaviour of complex ceramic materials, such as refractories. To the best of the authors knowledge, this is the first study investigating the applicability of CSLM in this research field. Traditionally, high-temperature refractory behaviour is studied through post-mortem microstructural characterisation of worn specimens (either industrially worn or from laboratory furnace tests, e.g. Refs. ^{25–29}). Although this methodology has been quite successful, it is essentially an indirect technique where the history of the sample has to be reconstructed from post-mortem analyses results. CSLM would, therefore, be an excellent complementary tool as it may provide direct, in situ information on complex solid state reaction mechanisms taking place at elevated process temperatures.

For the present feasibility study, a commercially available magnesia–chromite refractory type was selected as a ceramic test material. The reason for this choice is threefold. Firstly,

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at high-temperatures the two principal phases in magnesia–chromite refractories (periclase and magnesiochromite spinel) strongly interact with one another, hence making this material an interesting case-study for *in situ* observations. Secondly, considerable knowledge on these materials has already been obtained by the present authors through post-mortem investigations^{25–27,30}. Such studies may serve as a reference to compare the present CSLM observations. Finally, although environmental and economical restrictions motivate metallurgists to find chrome-free alternatives, for many demanding applications magnesia–chromite bricks remain the most suitable material currently commercially available. If the CSLM method proves to be successful for this complex ceramic material, then it can be extended to other ceramic systems.

2. Experimental methods and analysis

The experimental approach was to directly visualise the high-temperature behaviour of a complex refractory (ceramic) system with a CSLM. Particular zones in the refractory specimens were selected for thorough analysis. In order to allow for comparison, images were also obtained with light optical microscopy (LOM) and microprobe backscattered electron (BSE) images taken prior to and after the CSLM runs. Chemical and microstructural characterisation was performed with electron probe micro-analysis energy dispersive spectroscopy (EPMA-EDS). To evaluate the effect of high-temperature reactions between the distinct phases present in the system, roughness analyses were performed, also prior to and after the CSLM experiments.

2.1. CSLM

The experiments were performed with a confocal scanning laser microscope (Model ILM21H, Lasertec) equipped with a high-temperature cell (Model SVF17SP, Lasertec). For a detailed description of the technique, we refer to the work of Emi and colleagues.^{1,2} What follows here, is a summary of the CSLM essentials that are directly relevant for the present study. In the CSLM, He–Ne laser light (wavelength of 632.8 nm) is focussed by an objective lens onto the sample, with the reflected beam being focussed on a photon detector via a beam splitter. By scanning the focussed spot relative to the object an image can be constructed.

The CSLM technique operates with a confocal pinhole, which only permits light incident from the focal plane to pass through to the photon detector (Fig. 1). In contrast with conventional microscopy, in confocal optics the maximum intensity only occurs for the focussed points (Fig. 2). This implies that the axial resolution of the CSLM technique is high (i.e. short ‘focal depth’ or ‘depth of field’: ca. 0.2 μm for the $\times 100$ objective lens with a numerical aperture (NA) of 0.95: supplier data). For smaller magnifications (e.g. $\times 20$ objective lens) this focal depth is higher (see further). The pinhole also blocks the thermal radiation emitted from the specimen other than the focal plane. Hence, only the polarised reflection of the high intensity laser beam reaches the imaging sensor, thereby making it possible to observe samples at high lateral resolution (ca. 0.25 μm , for a

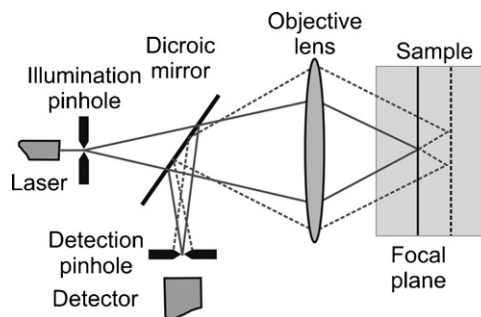


Fig. 1. Principle of confocal optics used in CSLM [modified from Ref. ³¹].

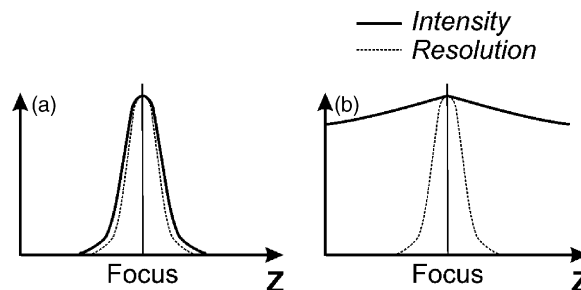


Fig. 2. Comparison between confocal (left) and conventional microscopy (right), with *Z* being the (vertical) distance from the objective lens.

$\times 100$ objective lens (NA = 0.95): supplier data) at elevated temperatures (theoretically up to 1700 °C with the present furnace setup).

Fig. 3 shows a schematic overview of the gold-plated paraboloid chamber (IIF), i.e. the high-temperature cell. The mirror polished refractory specimen (see further) is put into an alumina crucible (inner diameter = 5.9 mm, height = 4 mm), which in its turn is placed in a Pt sample holder equipped with a B-type thermocouple (PtRh). Fig. 4 shows the complete assembly, which is inserted in the hot zone of the high-temperature cell (upper focal point of the chamber). The heating occurs through

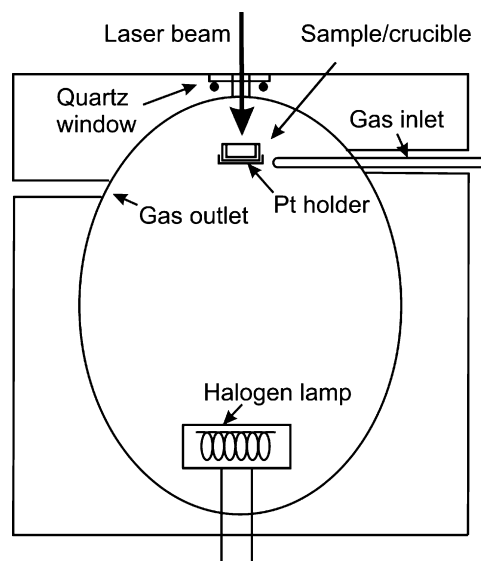


Fig. 3. Schematic of the high-temperature cell of the CSLM [modified from Ref. ¹⁴].

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