ADVANCED REAL TIME OPTICAL IMAGING

Advanced Real-Time Optical Imaging: Part I

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Materials research is significantly assisted by information gathered from imaging. Images may be macroscopic or microscopic; different imaging techniques ranging from conventional microscopes to synchrotron tomography are selected for various research purposes and goals. While most materials imaging is performed in static environments at room temperature because of its appropriateness (or due to device limitations), simple optical imaging may transform into a powerful tool if it is dynamically captured during materials treatments, processing, or physical/chemical exposures. Real-time observations this way provide researchers with vital information on materials behaviors under controlled but various conditions.

In the field of energy materials, it is not uncommon to require materials studies under high-temperature, high-pressure, and/or corrosive conditions. In gasifiers, for example, investigations of sensor alloy degradation, slag-refractory reactions, and coal agglomeration require samples to undergo severe environments where direct imaging would be difficult using conventional experimental methods. While post mortem analysis often provides critical information, interpretation may sometimes be complicated without confirming such occurrences under those conditions.

Advanced real-time optical imaging techniques enable in situ investigations on materials interactions, phase transformations, and physical variations in controlled environments, minimizing empirical uncertainties. Temporal and spatial understandings of behavioral mechanisms can then be extracted free from artifacts associated with

conventional experimental practices. The information that can be obtained from these techniques includes real-time thermodynamic and kinetic data reflecting a variety of materials and processes, which allows construction of phase diagrams and kinetic models.

Advanced real-time optical imaging devices may include thermal imaging furnaces, environmental confocal scanning laser microscopes, hot thermocouple and sessile drop imaging systems, etc. These techniques may be operated in situ, in operando, in vitro, and in vivo to produce dynamic imaging. Optical sources for those approaches are selected depending on their purpose; For example, while visible light is readily available, a laser with fixed wavelength may be preferred for observing high-temperature phenomena at high resolution. Characteristic information, in addition to imaging, may be collected if infrared or ultraviolet light is used. Enhanced imaging techniques in conjunction with additional devices can cogenerate other useful data including temperature, oxygen partial pressure, physical characteristics, resistivity, Raman spectrum, and gas composition to support visual variations from imaging.

Due to the importance of such techniques, significant efforts have been made by researchers and manufacturers to enable real-time materials observations in modern materials science. One noteworthy example is the environmental confocal scanning laser microscope (CSLM). The first commercial environmental CSLM appeared in 1986, being reflection based and utilizing a He–Ne laser with wavelength of 633 nm at scan rates up to 30 fps. With the pioneering efforts of Emi, Shibata, Yin et al. at Tohoku University (Yin now with ArcelorMittal)^{1,2} and Nakamura at Lasertec (now with Yonekura), substantial improvement of the heating chamber and microscope system was achieved, enabling real-time investigations of molten metals and slags at high temperatures in controlled atmospheres. Following the invention of blue light-emitting diodes (LEDs) in 1992, CSLMs

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became more economically affordable. Modern CSLMs can offer high resolution (140 nm), partly benefitting from an LED with wavelength of 405 nm, and high scan rates up to 120 fps.

Another representative imaging method, the hot thermocouple technique (HTT), was introduced by Kashiwaya, Cicutti, Cramb, and Ishii in 1998.³ This technique, utilizing an alternating current to control and record temperature, is applicable to make direct observations of materials under controlled temperatures and atmospheres. Crystal formation and growth in melts can be investigated at a single temperature or across a temperature gradient using this method.

The impact of emerging real-time imaging techniques in modern research can be discussed in the context of publication trends. The number of publications featuring real-time imaging techniques started to rise exponentially after 1990. According to Clarivate Analytics Web of Science⁴ (Analytics), the total of such papers published in 1990 was only 20 but grew rapidly to 3817 in 2017. These numbers are dominated by publications in the optics and bio and medical categories. If such analysis is narrowed to the metallurgical field, 48 papers were published in 2017 (compared with 1 in 1991). The number of citations in this field was 867 in 2017, giving an apparent citation per item of 18 in 2017 (compared with 1 in 1991). In the field of energy and fuels, 24 papers were published in 2017 (compared with none in 1991), while the number of citations was 628 in 2017, giving an apparent citation per item of 26 in 2017 (compared with none in 1991). 110 papers were published in the biomaterials field in 2017 (compared with 9 in 1994), while the number of citations in that year was 3771, yielding an apparent citation per paper of 34 (compared with 0.1 in 1994). Clearly, studies based on real-time imaging techniques have been playing significant roles in modern materials research.

In recognition of materials studies utilizing advanced imaging techniques, the symposium “Advanced Real-Time Optical Imaging” was organized at the TMS 2018 Annual Meeting and Exhibition in Phoenix, Arizona. During the symposium, the conference room was always filled with an enthusiastic audience, where invaluable papers from all over the world including Australia, Belgium, Canada, China, Japan, South Korea, Sweden, United Kingdom, and the United States of America were presented. Following this success, it was decided that the symposium should continue to be held at subsequent TMS meetings. The next “Advanced Real-Time Imaging” symposium will take place at the TMS 2019 Annual Meeting and Exhibition in San Antonio, Texas. Research using a variety of real-time imaging techniques is expected to be presented, sharing cutting-edge findings from these approaches.

The goal of this special *JOM* topic is to assemble the advanced real-time optical imaging techniques in a single venue, to emphasize the diversity and strength of such approaches. While these techniques have been widely used as enhanced tools to aid materials studies, such reports have been dispersed among different journals and conference sessions in the past. Papers were contributed to this topic by experts and pioneers in the field of real-time optical imaging, describing excellent insights and applications. Due to the high volume collected, the papers for this topic will be featured in two segments: part I in July and part II in October.

The following papers are published under the topic “Advanced Real Time Optical Imaging: Part I” in the July 2018 issue (vol. 70, no. 7) of *JOM* and can be accessed via the *JOM* page at <http://link.springer.com/journal/11837/70/7/page/1>:

Environmental confocal scanning laser microscopy:

- “Quantitative and Qualitative Aspects of Gas–Metal–Oxide Mass Transfer in High-Temperature Confocal Scanning Laser Microscopy” by Stephano P.T. Piva, P. Chris Pistorius, and Bryan A. Webler
- “In Situ Observations of Agglomeration of Non-metallic Inclusions at Steel/Ar and Steel/Slag Interfaces by High-Temperature Confocal Laser Scanning Microscopy: A Review” by Wangzhong Mu, Neslihan Dogan, and Kenneth S. Coley
- “A Review of In Situ Observation of Crystallization and Growth in High-Temperature Oxide Melts” by Zhanjun Wang and Il Sohn
- “In Situ Observation of Dissolution of Oxide Inclusions in Steelmaking Slags” by Mukesh Sharma, Wangzhong Mu, and Neslihan Dogan
- “Evolution of Near-Surface Internal and External Oxide Morphology During High-Temperature Selective Oxidation of Steels” by Mary E. Story and Bryan A. Webler
- “Structural Changes and Material Transport in Al₂O₃-Supported Cu/Fe Spinel Particles in a Simulated Chemical Looping Combustion Environment” by W.H. Harrison Nealley, Anna Nakano, Jinichiro Nakano, and James P. Bennett

Environmental optical microscopy:

- “In Situ Interface Observation of 3C-SiC Nucleation on Basal Planes of 4H-SiC During Solution Growth of SiC from Molten Fe–Si Alloy” by Sakiko Kawanishi and Takeshi Yoshika

Hot thermocouple technique:

- “High-Temperature Properties of Mold Flux Observed and Measured In Situ by Single/Double Hot-Thermocouple Technique” by Wanlin Wang, Peisheng Lyu, Lejun Zhou, Huan Li, and Tongsheng Zhang

Particle image velocimetry:

- “Validation of Physical Simulations of a Stirred Molten Metal Using Particle Image Velocimetry Data” by Agnieszka Dybalska, Dmitry G. Eskin, and Jayesh B. Patel

High-speed camera technique:

- “Improving Keyhole Stability by External Magnetic Field in Full-Penetration Laser Welding” by Min Li, Jiajun Xu, Yu Huang, and Youmin Rong

Infrared imaging:

- “Radiative Properties of Thin Films of Common Dielectric Materials in IR Spectral Range of 1.5–14.2 μm : Application to Infrared Imaging” by Asahel Bañobre, Sita Rajyalaxmi Marthi, and N.M. Ravindra

Review on multiple techniques:

- “Utilization of High-Temperature Slags From Metallurgy Based on Crystallization Behaviors” by Yongqi Sun and Zuotai Zhang

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3. Y. Kashiwaya, C. Cicutti, A. Cramb, and K. Ishii, *ISIJ Int.* 38, 348 (1998).
4. *Web of Science*. <https://webofknowledge.com>. Accessed 21 Feb 2018.