

Advanced Real Time Optical Imaging

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Materials research is greatly 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 to 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 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 postmortem analysis often carries critical information, interpretation may be sometimes complicated without confirming such occurrences under those conditions. The advanced real time optical imaging techniques enable in situ investigations on materials interactions, phase transformations, and physical variations in controlled environments, which minimizes empirical uncertainties. Temporal and spatial understandings of behavioral mechanisms can then be extracted free from artifacts associated with the 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.

The advanced real time optical imaging devices may include *thermal imaging furnaces, environmental confocal scanning laser microscopes, hot thermocouple techniques, sessile drop imaging techniques, 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 purposes. For example, while visible light is readily available, a laser with a fixed wave length may be preferred for observing high temperature phenomena at high resolution. Characteristic information, in addition to imaging, may be collected if infrared and ultra violet lights are used. The enhanced imaging techniques 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 would be the environmental confocal scanning laser microscopes (CSLMs). The first commercial environmental CSLM appeared in 1986, which was reflection-based, utilizing a He-Ne laser with a wavelength of 633 nm at a scan rate up to 30 fps. With pioneering efforts of Emi, Shibata, Yin, et al. at Tohoku University [1, 2] and Nakamura at Lasertec (now with Yonekura), the substantial improvement of the heating chamber and microscope system was achieved, enabling investigations of molten metals and slags at high temperatures in controlled atmospheres. Following the invention of blue LEDs in 1992,

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

Another representative imaging method, a hot thermocouple technique (HTT), was introduced by Kashiwaya, Cicutti, Cramb, and Ishii in 1998 [3]. The technique utilizing an alternating current to control and record temperature was intelligent to make direct observations of materials at controlled temperatures and atmospheres. Crystal formation and growth in melts may be investigated at a single temperature or across a temperature gradient using this method.

The impact of the emerging real-time imaging techniques in modern research can be discussed by respecting publication trends. The number of publications featuring real time imaging techniques started to drastically rise after 1990. According to Clarivate Analytics Web of Science [4], total such papers published in 1990 was only twenty, but it rapidly grew to 3,817 in 2017. The numbers are dominated by publications in the optics and bio and medical categories. If it is narrowed to the metallurgical field, 48 papers were published in 2017 (compared to one in 1991). Citations in this field in 2017 were counted to be 867, which gives an average (and apparent) citation per item to be 18 in 2017 (compared to one in 1991). In the field of energy and fuels, 24 papers were published in 2017 (compared to zero in 1991) while times cited in 2017 were recorded to be 628, which gives an average (and apparent) citation per item to be 26 in 2017 (compared to zero in 1991). 110 papers were published in the biomaterials field in 2017 (compared to 9 in 1994) while citations in this year were counted to be 3,771, which returns an average (and apparent) citation per paper to be 34 (compared to 0.1 in 1994). Clearly, studies based on real time imaging techniques have been increasingly proving their significance in modern research.

In recognition of materials studies utilizing the advanced imaging techniques, the symposium 'Advanced Real Time Optical Imaging' was organized at TMS 2018 in Phoenix, AZ. During the symposium, the conference room was always filled by an enthusiastic audience where invaluable papers from all over the world including Australia, Belgium, Canada, China, Japan, South Korea, Sweden, UK, and USA were presented. Following this success, it was decided that the symposium would continue to be held at the TMS meetings. The next symposium 'Advanced Real Time Imaging' will take place at TMS 2019 in San Antonio, TX. Research using a variety of real time imaging techniques is expected to be presented sharing cutting-edge finding from those tactics.

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 those techniques have been widely used as enhanced tools to aid materials studies, those utilizing them have been reported dispersedly over different journals and conferences in the past. The following papers were contributed from experts and pioneers in the field of real time optical imaging and provided excellent insights and applications for each practice:

Advanced Real Time Optical Imaging

'Environmental' confocal scanning laser microscopy:

- 'Quantitative and Qualitative Aspects of Gas-Metal-Oxide Mass Transfer in High-Temperature Confocal Scanning Laser Microscopy' – S. P.T. Piva, P. C. Pistorius

- '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' – W. Mu, N. Dogan, K. S. Coley
- 'A Review of In-Situ Observation of Crystallization and Growth in High Temperature Oxide Melts' – Z. Wang, I. Sohn
- 'In Situ Observation of Dissolution of Oxide Inclusions in Steelmaking Slags' – M. Sharma, N. Dogan, W. Mu
- 'In-situ Visualization of High Temperature Selective Oxidation' – M. E Story, B. A. Webler
- 'High temperature confocal laser scanning microscopy studies of ferrite formation in inclusion-engineered steels: a review' – W. Mu, P. Hedström, H. Shibata, P. G. Jönsson, K. Nakajima

'Mechanical' confocal scanning laser microscopy:

- 'Use of confocal microscope for environmental tensile mechanical testing' – K. Rozman, W. H. H. Nealley, J. Nakano, S. Akanda, Ö. N. Dogan, J. A. Hawk
- 'Assessing Mechanical Behaviors of Additive Manufactured Alternating Layers of Inconel-718/H13 Tool Steel' – B. E. Tucker, W. H. H. Nealley, B. Dutta, J. Nakano, and D. Maurice

'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' – S. Kawanishi, T. Yoshikawa

Hot thermocouple technique:

- 'High-temperature Properties of Mold Flux in-situ Observed and Measured by Single/Double Hot Thermocouple Technique' – W. Wang, P. Lyu, L. Zhou, T. Zhang

Particle image velocimetry:

- 'Validation of the Physical Simulations of a Stirred Molten Metal Using Particle Image Velocimetry Data' – A. Dybalska, D. Eskin, J. Patel

High speed camera technique:

- 'In-situ investigation of slag-metal interactions in LD converter upon low voltage application' – S. Semykin, T. Golub
- 'Improving Keyhole Stability by External Magnetic Field in Full Penetration Laser Welding' – M. Li, J. Xu, Y. Huang, Y. 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' – A. Bañobre, S. R. Marthi, N. M. Ravindra

Review on multiple techniques:

- 'Utilization of High-Temperature Slags from Metallurgy Based on Crystallization Behaviors' – Y. Sun, Z. Zhang

JOM Technical Communications

- 'Structural Changes and Material Transport in Al₂O₃-supported CuFe₂O₄ Particles in a Simulated Chemical Looping Combustion Environment' – W. H. H. Nealley, A. Nakano, J. Nakano, J. P. Bennett

References

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3. Kashiwaya;, Y., et al., *Development of Double and Single Hot Thermocouple Technique for in Situ Observation and Measurement of Mold Slag Crystallization*. ISIJ International, 1998. **38**(4): p. 348-356.
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