European Winter Conference on Plasma Spectrochemistry

Ljubljana, Slovenia,
January 29th – February 3rd, 2023

Book of Abstracts

NATIONAL INSTITUTE OF CHEMISTRY

EWCPS

Book of abstracts of the 19th European Winter Conference on Plasma Spectrochemistry

Publisher:
National Institute of Chemistry
Hajdrihova 19
1000 Ljubljana
Slovenia

Editors:
Vid Simon Šelih and Martin Šala, National Institute of Chemistry, Slovenia

Graphical design:
Repster

Ljubljana, January 2023
Publication is not for sale

Kataložni zapis o publikaciji (CIP) pripravili v Narodni in univerzitetni knjižnici v Ljubljani
COBISS.SI-ID 139103747
ISBN 978-961-6104-85-2 (PDF)

Implementation of a new heating stage for in-situ LIBS analysis of temperature induced processes

Jakob Willner[1], Maximilian Podsednik[1], Birgit Achleitner[2], Tobias Huber[2,3], Michael Nelhiebel[1], Silvia Larisegger[1], Andreas Limbeck[2]

[1]KAI Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Villach, Austria, [2]TU Wien, Institute of Chemical Technologies and Analytics, Vienna, Austria, [3]Huber Scientific, Vienna, Austria

Temperature-induced and temperature-dependent processes in solid materials are vital for many technological application fields. In related research, samples are often treated in an oven at a defined temperature for a specific time and analyzed ex-situ after cooling the sample to ambient temperature. This approach can be prone to errors for multiple reasons. The observed process can continue further during cooling, and rapid cooling could change the sample properties and lead to stress or cracks. Moreover, further reactions can occur if the sample-surrounding atmosphere is not controlled (humidity, traces of corrosive gases, oxygen). Thus, for concerning applications, in-situ analysis is indispensable.

Laser Induced Breakdown Spectroscopy (LIBS) is a powerful technique for direct solid analysis, offering elemental information of the entire periodic table and the possibility of spatially resolved measurements - analytical capabilities which make LIBS perfectly suited for the characterization of processed, aged or degraded samples. However, commercial LIBS instruments only allow operation at ambient temperature, permitting solely the ex-situ investigations mentioned before.

This work presents the development and application of a newly implemented heating stage for in-situ LIBS, enabling measurements at temperatures up to 1000 °C while the chamber atmosphere can be changed as required. This combination allows the investigation of a large variety of scientific questions.

The versatile applicability is demonstrated in different examples by the in-situ acquisition of time-resolved results. The scaling of copper under synthetic air was monitored at different temperatures by measuring the oxygen signal of the growing oxide layer. Exploiting the capability of LIBS to obtain also molecular emission, e.g., C2 or CN, the imidization reaction of precursers to a polyimide film was monitored at different temperatures. Finally, the interdiffusion of metals in layered systems was investigated using depth-resolved analysis.

in-situ LIBS, heating stage, material characterization