Montanuniversität Leoben

Integrated Multi-Modal, Multi-Scale Characterization of Complex Slag Systems

An Orthogonal Analytical Framework Combining Raman Spectroscopy (RAMAN), X-ray Diffraction (XRD), Scanning Electron Microscopy with Energy-Dispersive X-ray Analysis (SEM-EDX), and Transmission Electron Microscopy (TEM)

Cluster of Excellence **Recycling**

Green Zinc develops CO_2-neutral, zero-waste routes for recovering valuable metals from secondary resources by replacing fossil coke with renewable reductants and by valorising all by-products through targeted slag conditioning. Detailed characterization enables tuning of slag composition and cooling/conditioning to immobilize hazards and engineer application-ready properties.

Context, Objectives, and Analytical Approach

Slag from lead production and steel mill dust from electric arc furnace steelmaking are complex residues, rich in metal oxides (Fe, Zn, Pb) and silicates. Untreated, they pose leaching risks, yet they also represent valuable secondary resources whose recovery and reuse align with the objectives of Green Zinc to achieve CO_2 neutral, zero waste metallurgy. The central question is how these materials can be treated for maximizing valuable metal recovery and for a safe utilization of the generated slag. This depends on understanding how composition and cooling conditions govern phase formation, microstructure, stability, leach resistance, and mechanical performance. To realize the objectives of Green Zinc and deliberately tailor the properties of pyrometallurgically reduced slags and dust derived slags for further use, comprehensive characterization is essential to identify all relevant and controllable parameters including phase assemblage, glass and crystal fraction, microchemistry, morphology, and grain size and shape. This motivates an orthogonal multi scale analytical strategy that integrates complementary techniques to deliver a coherent picture from bulk to micro and nano scales, translating measurements into actionable levers such as composition, additives, cooling and conditioning for robust property engineering and safe utilization, introduced in the following methods.

Orthogonal Analytical Framework

SEM-EDX

SEM provides high contrast images of microstructure, while EDX adds elemental composition at points and over maps. Together they deliver morphology, grain size information, identification of inclusions and interfaces, and element partitioning across phases for key elements such as Fe, Zn, and Pb. These outputs link processing to structure and chemistry, showing where metals reside, how they are immobilized, and which features may control mechanical properties and leach resistance.

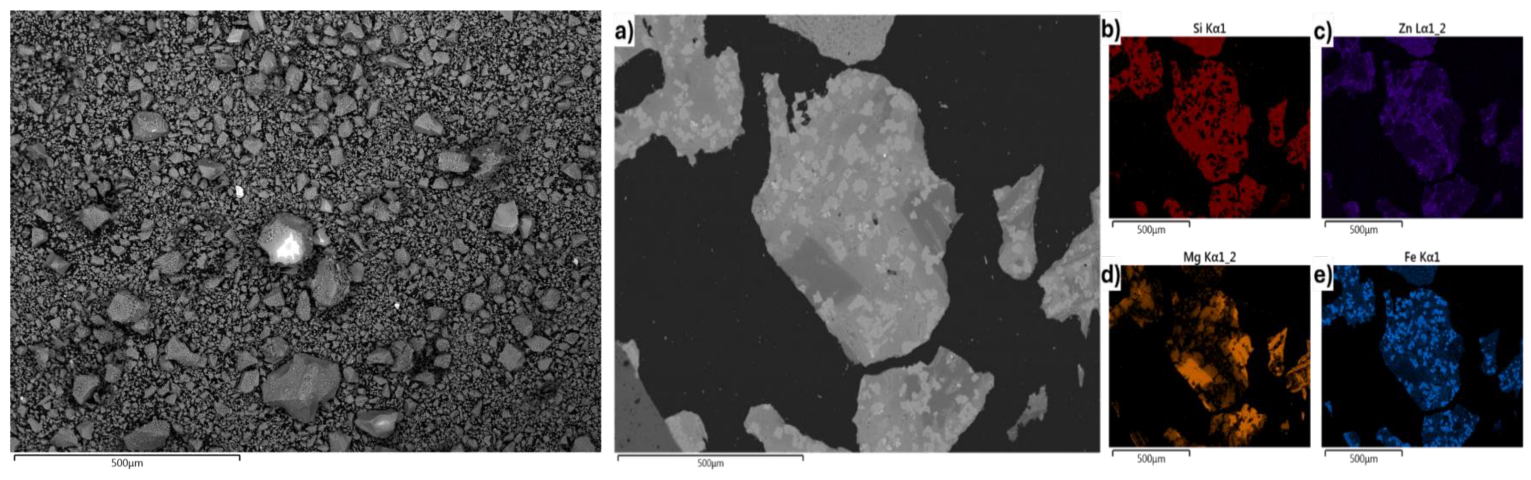

Figure 1: SEM–BSE image of the lead slag with corresponding EDX maps of the dominant elements; scale bars as indicated.

XRD

XRD identifies the crystalline phases present in the slag and separates them from any glassy fraction through their diffraction patterns. It delivers a phase list with confidence, an estimate of relative phase amounts through quantitative refinement, and indicators of overall crystallinity versus glass. These outputs provide the baseline for process control, confirming whether the targeted mineral assemblage has been achieved and whether metals are tied up in thermodynamically stable crystal lattices.

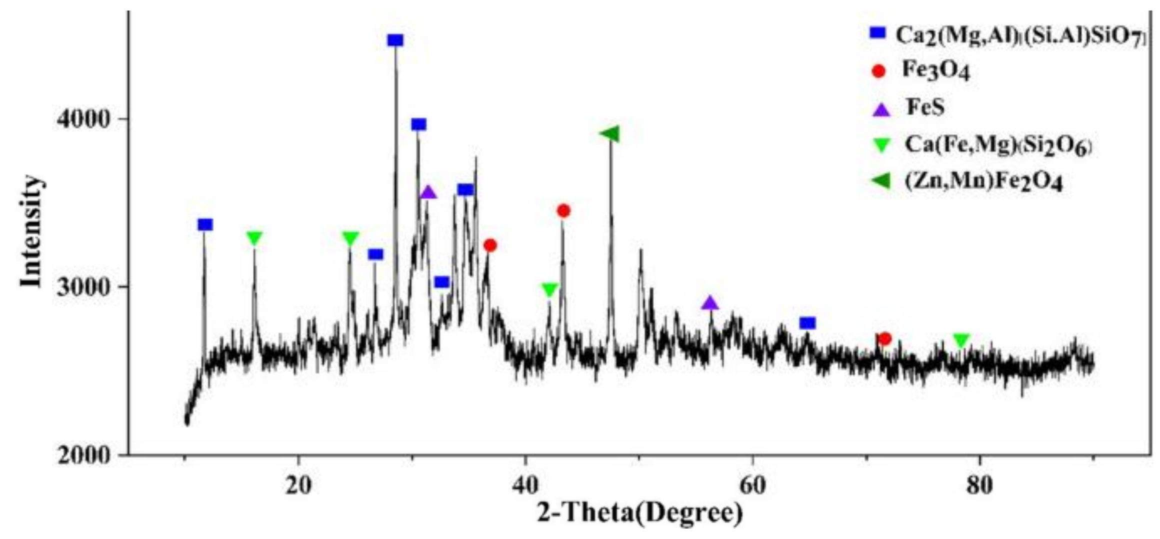

Figure 2: XRD pattern of steel mill dust slag with reference phase markers.

RAMAN

Raman probes local molecular structure and complements XRD by detecting poorly crystalline and amorphous constituents as well as minor phases. It delivers point spectra and maps that confirm phase assignments directly on polished sections, visualize the spatial distribution of phases, and indicate the balance between crystalline domains and the silicate glass network. These outputs verify phase calls at the microscale and reveal heterogeneity that is relevant for leaching behaviour and downstream performance.

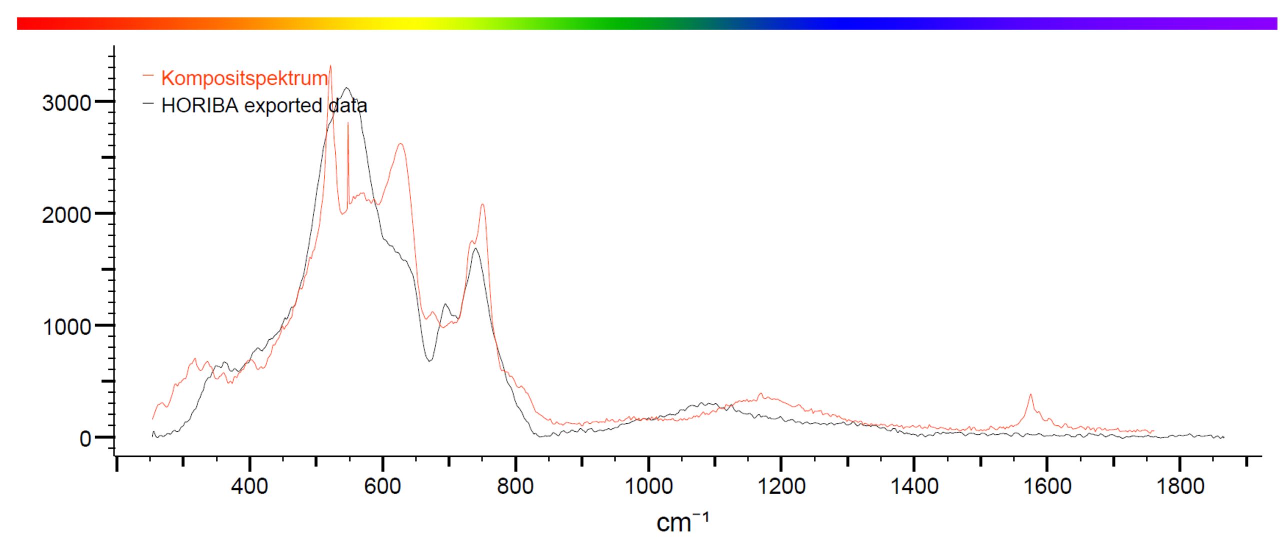

Figure 3: Raman of Pb-slag: measured (red) vs. melilite (akermanite–gehlenite) reference (black).

TEM

TEM examines the slag at nanometer scale using high resolution imaging, selected area electron diffraction and STEM-EDS to resolve crystal lattices, nanoscale precipitates and glass and crystal interfaces. It delivers identification of phases below 100 nanometers, estimates of crystallite size and defect density, and interface chemistry. These outputs complement XRD, Raman and SEM by providing nanoscale evidence that links local structure to leaching resistance and mechanical performance.

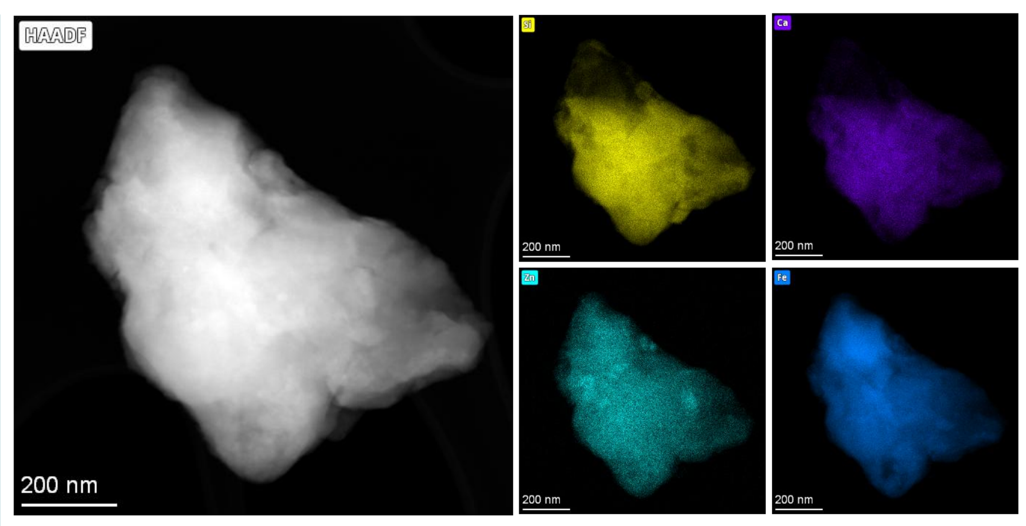

Figure 4: High-Angle Annular Dark-Field (HAADF) STEM image of the slag

Contact

Dipl. Ing. Ehab Tara
Department Metallurgy
Chair of Nonferrous Metallurgy
ehab.tara@unileoben.ac.at
+43 3842 402 5264
www.nichteisenmetallurgie.at
www.greenzinc.at

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