Analyzing Spinel refractory MgO-Al_2O_3 premix using ARL X'TRA Companion XRD

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Introduction

Spinel refractories—primarily magnesia–alumina ($MgAl_2O_4$)—are the workhorses of high-temperature linings in steel ladles, cement rotary kilns, glass tanks, and non-ferrous smelters. Their appeal lies in a combination of shock resistance, chemical stability against basic slags, and an expansion behavior that mitigates cracking. Depending on the duty, they are used as bricks, castables, or gunned repairs, sometimes with engineered microstructures that blend spinel with periclase, corundum, or zirconia to tune creep, corrosion, and spalling performance.

X-ray diffraction (XRD) underpins manufacturing control from raw mix to fired product. During synthesis, XRD tracks the solid-state reaction of MgO and Al_2O_3 into spinel, verifying the disappearance of periclase and alumina and quantifying the spinel fraction by Rietveld analysis. Lattice parameters reveal cation stoichiometry and solid-solution effects; peak breadth trends indicate crystallite growth and microstrain, proxies for sintering progress. In finished components, XRD detects secondary phases such as calcium aluminates introduced by binders, slags, impurities, and it provides fingerprinting for batch-to-batch consistency after thermal exposure. Embedded into routine QA/QC, XRD shortens feedback loops, stabilizes recipes, and links processing conditions to the microstructure that governs refractory life.

Figure 1: ARL X'TRA Companion X-ray diffraction system.

Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion X-Ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the state-of-the art solid state pixel detector (55x55 µm pitch), the ARL X'TRA Companion provides very fast data collection and comes with single-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System) seamless integrated into Thermo Scientific™ SolstiX™ Pronto instrument control software.

Experimental

A MgO/Al_2O_3 pre-mix sample was manually pressed in top loading sample cup and measured in reflection mode using Cu Kα (1.541874 Å) radiation for 10 minutes with sample spinning. (Figure 2). Rietveld refinements were carried out using Profex software [1].

Results and discussion

Quantitative XRD of the magnesia–alumina premix indicates a periclase-rich, in-situ spinel system with 43.9 wt% MgO, 33.0 wt% α-Al_2O_3, and 1.3 wt% $MgAl_2O_4$ already formed. From the corundum content, the mix has a spinel potential of ~46 wt% on firing, leaving ~31 wt% residual MgO to provide basicity and high refractoriness. The main impurity family is Ca–Si silicates—Merwinite (1.0 wt%), Åkermanite (1.2 wt%), Wollastonite (1.6 wt%), plus Quartz/Mullite (≈2.6 wt%)—consistent with natural DBM (Dead burned magnesia) and Bauxite sources and indicative of mid-grade purity rather than premium fused feed. Carbonates (Dolomite 5.6 wt%, Calcite 0.4 wt%, Magnesite 0.5 wt%) and Hydrates/Sulfates (Brucite 0.2 wt%, Doyleite 0.9 wt%, Kieserite 0.9 wt%, Epsomite 0.6 wt%) together contribute ~9 wt% LOI, supporting the need for pre-drying or pre-firing to avoid explosive spalling and to initiate spinel growth. Fe oxides / Spinels (magnetite 0.6 wt%, Magnesioferrite 0.7 wt%) are within normal trace ranges; anatase ~0.5 wt% fingerprints bauxite-derived alumina. Minor phyllosilicates (Kaolinite/Celadonite ≈ 2.1 wt%) likely reflect clay carryover and may slightly depress hot strength. Overall, the phase balance matches a serviceable $MgO–Al_2O_3$ premix suitable for carbon-free basic linings (e.g., cement/lime kilns, tundish work linings), provided moisture control and a firing schedule are used to convert corundum + MgO to spinel and to remove volatiles.

Your benefits

The ARL X'TRA Companion XRD workflow delivers answers in minutes, quantifying MgO, α-Al_2O_3, and $MgAl_2O_4$ while resolving Ca–Si silicates and trace Fe-spinels. Subtle differences between corundum and spinel are cleanly separated, and LOI drivers—carbonates, brucite, and Mg-sulfates—are identified and trended. These insights let you compute spinel potential, anticipate pore-closing expansion, and set the right pre-dry / prefire schedule to avoid spalling. You can clearly distinguish mid-grade, ore-derived feeds from premium low-silica materials, tighten incoming QA, and optimize formulations for cement/lime kilns, tundish linings, and other carbon-free basic applications.

[1] N. Döbelin, R. Kleeberg,
J. Appl. Crystallogr. 2015, 48, 1573-1580.

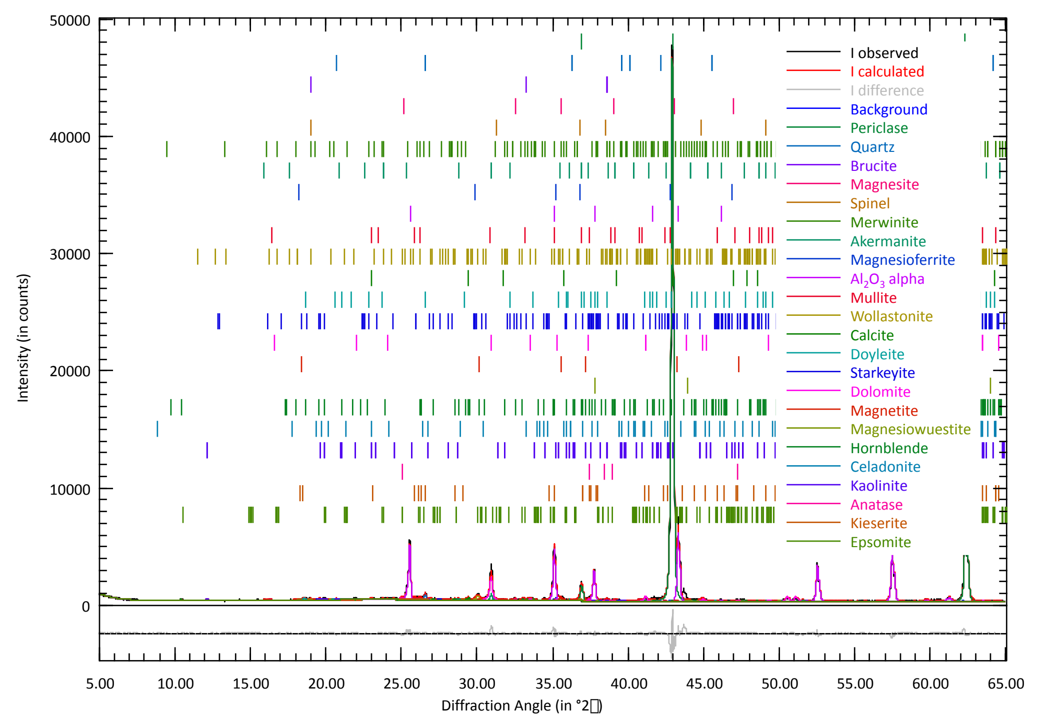

Figure 2: Measurement (10 minutes) of a MgO / Al_2O_3 pre-mix sample.

Table 1: Phase quantities from a Rietveld refinement of the pre-mix sample

Phase	Formula	Quantity (wt%)
Major phases		
Periclase	MgO	44.0
Corundum (α-Al_2O_3)	Al_2O_3	33.0
Ca–Si silicates (impurity family)		
Mullite	$Al_6Si_2O_{13}$	2.1
Wollastonite-2M	$CaSiO_3$	1.6
Åkermanite	$Ca_2MgSi_2O_7$	1.2
Merwinite	$Ca_3Mg(SiO_4)_2$	1.0
Quartz	SiO_2	0.5
Carbonates & hydrates (LOI contributors)		
Dolomite	$CaMg(CO_3)_2$	5.6
Doyleite	$Al(OH)_3$	0.9
Kieserite	$MgSO_4 \cdot H_2O$	0.9
Epsomite	$MgSO_4 \cdot 7H_2O$	0.6
Magnesite	$MgCO_3$	0.5
Calcite	$CaCO_3$	0.4
Brucite	$Mg(OH)_2$	0.2
Spinel & Fe-oxide family		
Spinel (magnesia–alumina)	$MgAl_2O_4$	1.3
Magnesioferrite	$MgFe_2O_4$	0.6
Magnetite	Fe_3O_4	0.6
Magnesiowüstite	$(Mg,Fe)O$	0.3
Phyllosilicates / amphibole traces		
Kaolinite	$Al_2Si_2O_5(OH)_4$	1.6
Hornblende*	$Ca_2(Mg,Fe)_4Al(Si_7Al)O_{22}(OH)_2$	1.1
Celadonite*	$K(Mg,Fe^{2+})(Fe^{3+},Al)(Si_4O_{10})(OH)_2$	0.5
Ti-bearing trace		
Anatase	TiO_2	0.5

* Representative idealized formulas shown for solid-solution minerals.

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