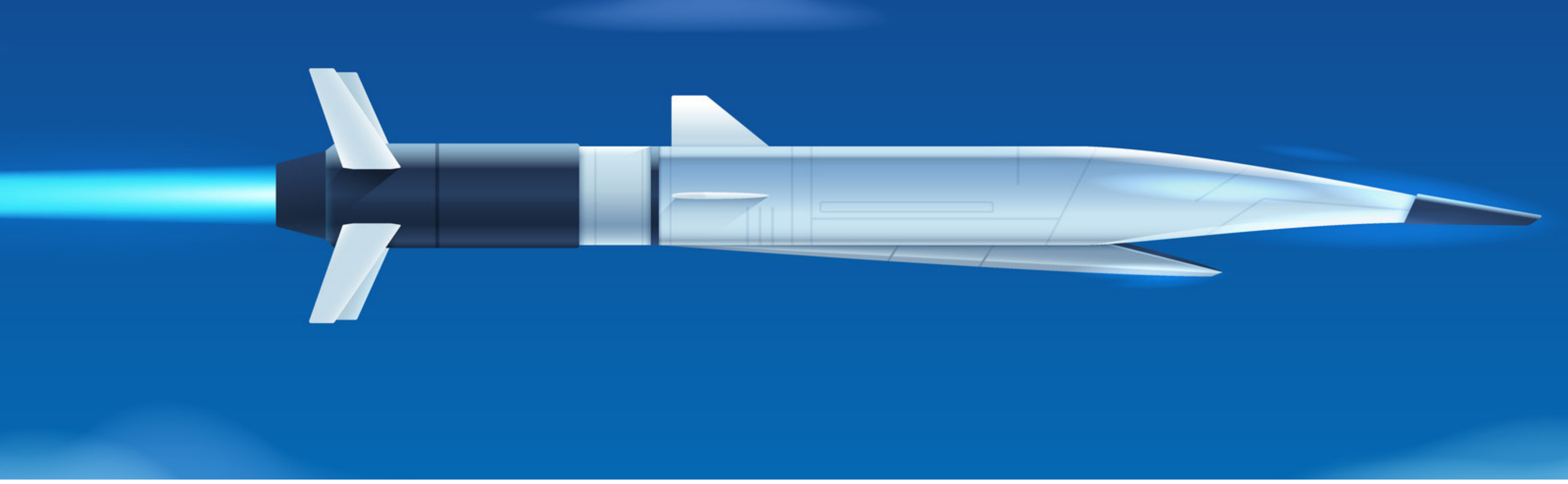

Analyzing titanium carbide ceramics: From tooling to hypersonic applications using ARL X'TRA Companion XRD

Authors

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

Introduction

Titanium carbide (TiC) is one of the hardest known ceramic materials, combining extreme hardness, high melting point, and excellent chemical stability with electrical and thermal conductivity. These properties make TiC indispensable across a wide spectrum of advanced technologies—from wear-resistant tooling and protective coatings to composite reinforcements and ultra-high temperature ceramics for hypersonic flight. Understanding the exact phase composition, crystallinity, and defect structure is critical, as even small variations in stoichiometry, oxidation state, or lattice strain can strongly influence toughness, oxidation resistance, and thermal performance. X-ray diffraction (XRD) provides the essential means to characterize TiC, distinguishing it from related carbides and nitrides, quantifying crystallite size and strain, and detecting surface oxides or carbon-rich by-products. Such structural insights enable direct correlations to performance in demanding applications, whether ensuring reliable machining tools, improving armor systems, or fine-tuning thermal protection for aerospace vehicles.

Instrument and 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 XRD 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 XRD 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

Two (blue-gray and black color) TiC samples were manually pressed in top loading sample cups 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 phase analysis of the two TiC powders shows clear differences in composition and microstructural quality. The "blue" sample consists of 57.3 wt% TiC, accompanied by a high fraction of rutile (37.7 wt%) and minor TiN (4.1 wt%). This high oxide content indicates substantial surface oxidation, consistent with the observed blue–grey appearance of the powder. The refined values suggest that the TiC fraction is significantly degraded, and the material would require further treatment (e.g., carbothermal reduction) before use in structural or high-temperature applications.

In contrast, the "black" sample contains a much higher TiC fraction of 83.0 wt%, with only 5.6 wt% rutile, 4.6 wt% anatase, and 6.7 wt% TiN. The reduced oxide fraction and higher TiC content explain its darker color and suggest a more suitable starting material for applications such as cermets, coatings, or bulk sintering. The presence of TiN is beneficial in this context, as it can enhance hardness and oxidation resistance.

Taking together, these results confirm that the blue batch is heavily oxidized TiC feedstock, requiring reduction before further processing, while the black batch represents a TiC-rich, low-oxide material that can be directly employed in demanding applications. The clear distinction between the two highlights the importance of quantitative XRD in assessing TiC powder quality and suitability for tooling, coating, or ultra-high temperature ceramic development.

Your benefits

The ARL X'TRA Companion XRD system delivers results within minutes, enabling quantification of TiC alongside oxide and nitride secondary phases, which is difficult to determine using elemental analyses techniques like XRF. Subtle differences between rutile, anatase, and TiN are resolved. These insights make it possible to clearly distinguish between powders requiring reduction treatment and high-quality starting materials, supporting the efficient production of advanced TiC ceramics for both industrial and aerospace applications.

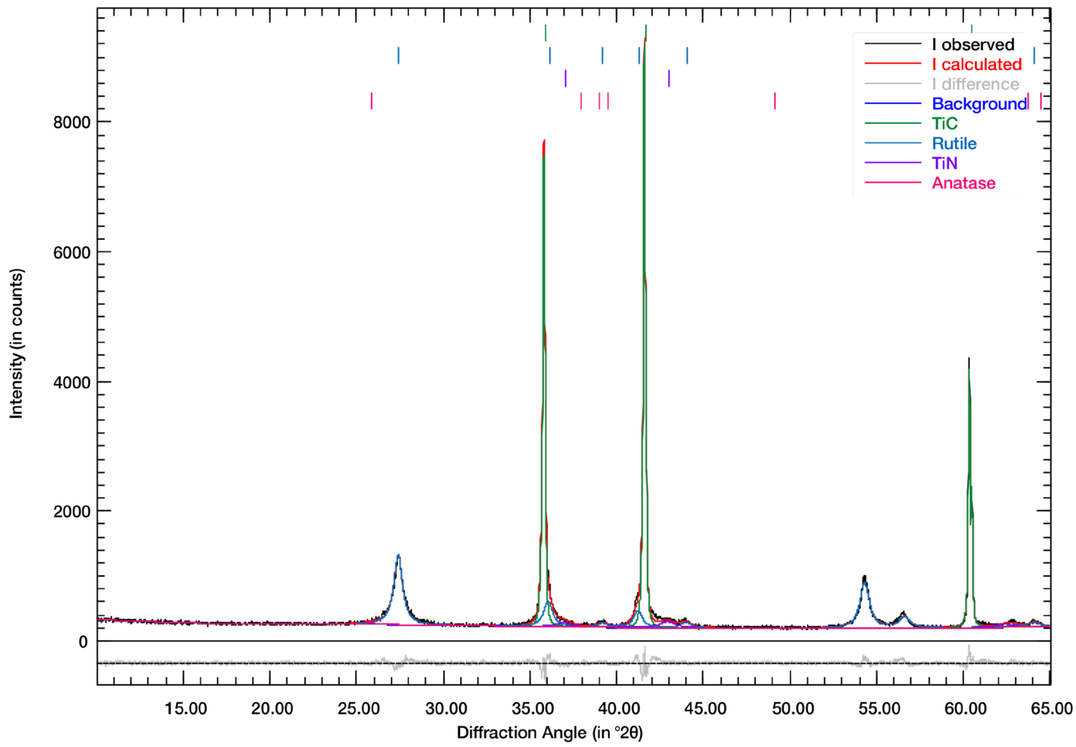

Figure 2. Measurement (10 minutes) of the blue TiC sample

Table 1. Phase quantities and crystallite size (CS) from a Rietveld refinement of the blue TiC sample

Phase	Formula	CS (in nm)	Quantity (in wt%)
TiC	TiC	172	57.3
Rutile	TiO2	14	37.7
Anatase	TiO2	8	0.9
TiN	TiN	8	4.1

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

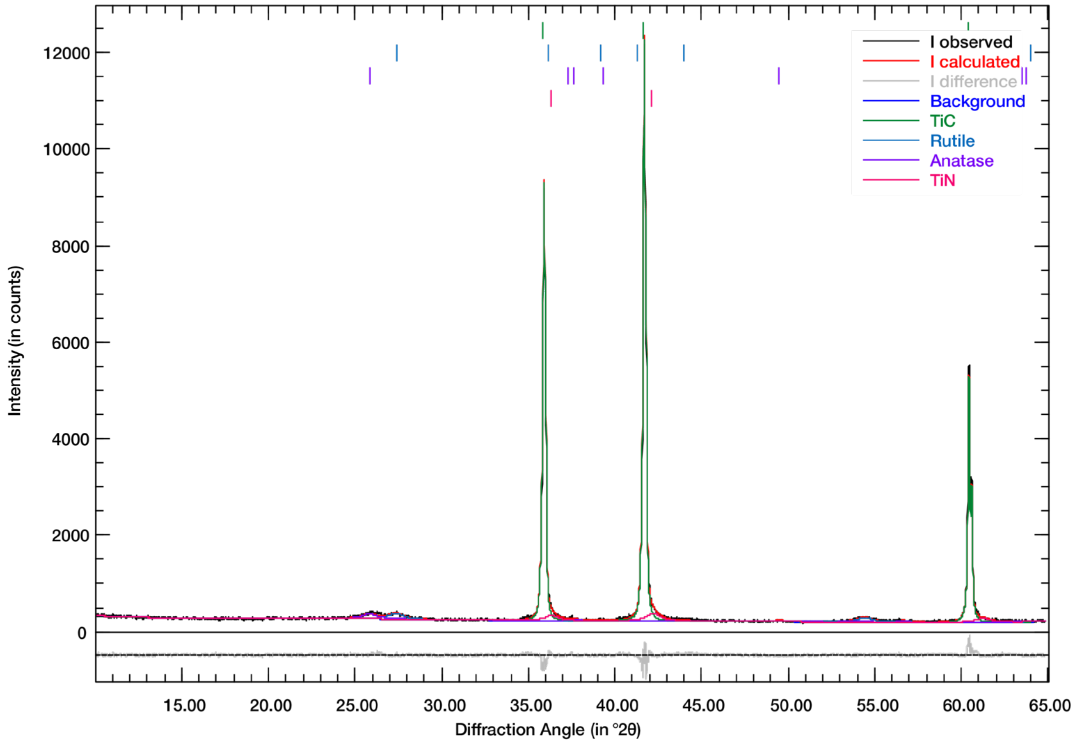

Figure 3. Measurement (10 minutes) of the black TiC sample.

Table 2. Phase quantities and crystallite size (CS) from a Rietveld refinement of the blue TiC sample

Phase	Formula	CS (in nm)	Quantity (in wt%)
TiC	TiC	137	83.0
Rutile	TiO2	8	5.6
Anatase	TiO2	7	4.6
TiN	TiN	8	6.7