



# Color Etching as a Tool for Phase Identification in an AA1100–25 Vol.% B<sub>4</sub>C Metal Matrix Composite

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Received: 18 October 2025 / Revised: 18 February 2026 / Accepted: 8 March 2026  
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## Abstract

A color-etching methodology was applied to an AA1100–25 vol.% B<sub>4</sub>C composite to enhance phase identification under optical microscopy. A solution of 50% (v/v) phosphoric acid in deionized water was used at –5 °C under a constant current density of 0.02 A cm<sup>–2</sup>. As a result, the aluminum matrix appeared pink, the B<sub>4</sub>C particles dark brown, TiB<sub>2</sub> interfacial layers formed distinct contours around the B<sub>4</sub>C reinforcement, and the Si-rich inclusions exhibited a bright blue hue. These contrasts made the interfaces between the B<sub>4</sub>C particles and the aluminum matrix clearly distinguishable. Complementary SEM and EDS analyses confirmed the presence of TiB<sub>2</sub> at the Al/B<sub>4</sub>C interfaces and Si-rich phases within the matrix. The proposed methodology provides a rapid and reliable means of identifying secondary phases and interfacial layers in Al–B<sub>4</sub>C composites.

**Keywords** Color Etching · Al–B<sub>4</sub>C composite · Phase Identification · Microstructural characterization

## Introduction

Aluminum–boron carbide (Al–B<sub>4</sub>C) metal matrix composites (MMCs) are widely employed as neutron-absorbing materials in the nuclear industry, combining the high neutron-capture cross-section of <sup>10</sup>B with the low density and excellent thermal conductivity of aluminum [1, 2]. Their applications include shielding and spent-fuel storage systems [1, 3, 4].

Previous studies on Al–B<sub>4</sub>C composites have focused on mechanical properties [5–12], corrosion resistance [13–21], or processing routes such as stir casting and powder metallurgy [22, 23]. However, few have examined metallographic preparation and optical microscopy, which remain challenging because of the hardness contrast and distinct polishing behavior between the ceramic and metallic phases [24, 25].

Reactions between molten aluminum and B<sub>4</sub>C can lead to the formation of interfacial compounds such as Al<sub>4</sub>C<sub>3</sub> and Al<sub>3</sub>BC, which degrade composite performance [26]. Titanium additions (1–2 wt.%) are commonly employed to

promote the formation of a TiB<sub>2</sub> layer that stabilizes the Al/B<sub>4</sub>C interface and limits interfacial reactions [22, 27]. In recycled alloys, Si-containing impurities may also appear as secondary phases [28], and conventional etching methods are often ineffective in revealing these phases under optical microscopy [29]. In this context, the present work proposes a color-etching method designed to distinguish the aluminum matrix, B<sub>4</sub>C reinforcement, TiB<sub>2</sub> interfacial layer, and Si-rich inclusions through color contrast.

## Experimental Procedure

An AA1100–25 vol.% B<sub>4</sub>C composite, commercially referred to as Boralcan™, was examined in this study. Manufactured by Rio Tinto Alcan for neutron shielding applications within the nuclear industry [30], Boralcan™ comprises an AA1100 aluminum matrix [31] reinforced with boron carbide (B<sub>4</sub>C) particles at volume fractions typically between 16 and 28 vol.% [32]. The specific composite utilized herein contains 25 vol.% B<sub>4</sub>C. Production involves direct-chill (DC) casting followed by hot rolling to yield dense plates exhibiting a uniform dispersion of the ceramic phase [33]. During processing, B<sub>4</sub>C powder is introduced into molten aluminum alloy, ensuring effective wetting and homogeneous particle distribution prior to solidification.

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Additionally, a minor titanium content (1–2.5 wt%) is incorporated to improve castability and bolster interfacial stability between the  $B_4C$  and liquid aluminum, thereby reducing the formation of brittle reaction products [32]. The detailed chemical composition of the analyzed composite is provided in Table 1.

The composite specimens were sequentially ground using SiC abrasive papers with grit sizes of 100, 220, 320, 400, 600, 800, and 1200. After grinding, the samples were polished using alumina suspensions with particle sizes of 1.0 and 0.5  $\mu\text{m}$ , followed by a final polishing step with 0.025  $\mu\text{m}$ . For the color-etching procedure, the specimens were electrolytically etched in a 50% (v/v) phosphoric acid ( $H_3PO_4$ ) aqueous solution at  $-5^\circ\text{C}$  under a constant current density of  $0.02\text{ A cm}^{-2}$ , with an aluminum counter-electrode. Both polished (unetched) and color-etched specimens were examined using an optical microscope (Leica). The polished

specimens were observed in bright field, whereas the color-etched specimens were examined under polarized light with a  $\lambda$ -plate; the analyzer was set to  $55^\circ$  and  $65^\circ$ . Scanning electron microscopy (SEM) analyses were carried out using a Hitachi TM3000 tabletop SEM equipped with energy-dispersive x-ray spectroscopy (EDS) operating in backscattered electron (BSE) mode, and a field-emission SEM (Phenom Pharos G2) for high-resolution imaging using a combined in-lens BSE–SE detection mode. Different accelerating voltages were used according to the analysis requirements, with higher voltages applied for EDS mapping and lower voltages employed to reduce interaction volume and enhance surface sensitivity.

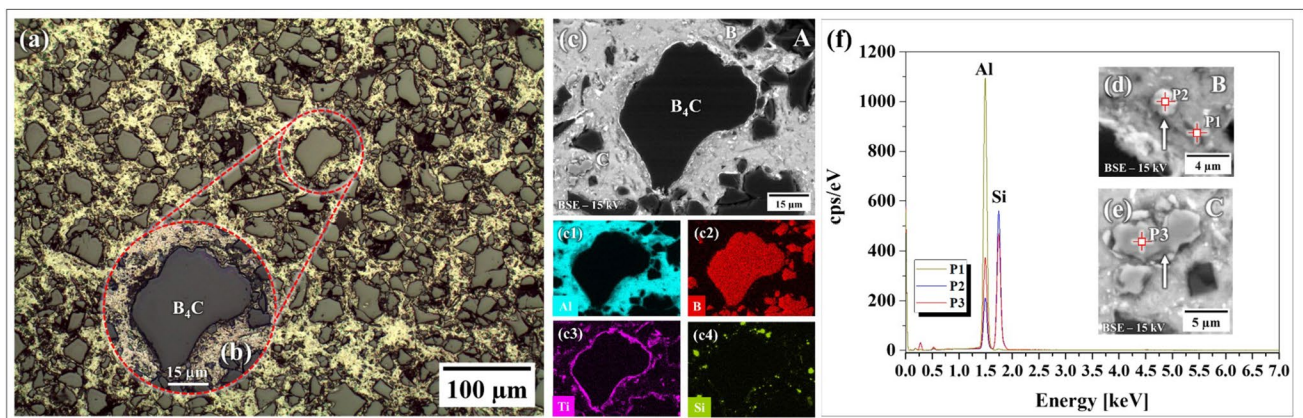
## Results and Discussion

The microstructure of the AA1100–25 vol.%  $B_4C$  composite is shown in Fig. 1. In the optical micrograph (Fig. 1a), the polished surface exhibits a uniform dispersion of angular  $B_4C$  particles throughout the aluminum matrix. A distinct color contrast allows the phases to be clearly distinguished: the  $B_4C$  particles appear dark gray, while the aluminum matrix displays a yellowish tone. The particles are irregular and angular, characteristic of  $B_4C$  powders that preserve their brittle morphology during the composite fabrication process [27]. At higher magnification (Fig. 1b), a single  $B_4C$  particle is shown in detail, revealing more clearly the morphology of the interface between the ceramic reinforcement and the metallic matrix.

The backscattered electron image as shown in Fig. 1c corresponds to the same particle indicated in Fig. 1b. The  $B_4C$

**Table 1** Chemical composition (wt%) of the AA1100–25 vol.%  $B_4C$  composite.

| Element      | wt%   |
|--------------|-------|
| B            | 18.1  |
| C            | 5.5   |
| Co           | <0.01 |
| Cu           | 0.10  |
| Fe           | 0.32  |
| Mn           | <0.01 |
| Mg           | <0.01 |
| Si           | 0.18  |
| Ti           | 2.6   |
| Zn           | <0.01 |
| Al (balance) | –     |



**Fig. 1** **a** Optical micrograph of the polished AA1100–25 vol.%  $B_4C$  composite; **b** Higher magnification of the dashed region in **a**, showing a  $B_4C$  particle embedded in the aluminum matrix; **c** Backscattered electron (BSE) image of the same  $B_4C$  particle observed in **b**; **c1–c4** EDS elemental maps corresponding to the region shown in **c**, highlighting the distribution of Al (cyan), B (red), Ti (magenta), and

Si (green); **d** and **e** Higher magnification BSE images of regions B and C indicated in **c**. The white arrows indicate Si-containing particles, while P1 and P2 mark the points where EDS spot analyses were performed. **f** EDS spectra corresponding to the analyses performed at points P1, P2, and P3. All SEM and EDS analyses were conducted at an accelerating voltage of 15 kV.

reinforcement appears darker than the surrounding matrix due to its lower average atomic number, which reduces the backscattered electron intensity compared with aluminum. This compositional contrast clearly delineates the boundary between the  $B_4C$  particle and the aluminum matrix. The corresponding EDS elemental maps (Fig. 1c1–c4) reveal the distribution of aluminum (cyan), boron (red), titanium (magenta), and silicon (green). Aluminum is concentrated in the matrix (Fig. 1c1), whereas boron is confined to the  $B_4C$  reinforcement (Fig. 1c2). Although boron is a light element with a low x-ray yield, its high concentration within the  $B_4C$  particle enhances the detected signal, allowing reliable identification with acceptable spatial resolution [33]. The titanium map (Fig. 1c3) shows a thin, continuous region surrounding the  $B_4C$  particle, attributed to the formation of a  $TiB_2$  layer that improves wetting and interfacial stability during solidification [22, 27]. Silicon is also detected (Fig. 1c4) as fine, bright particles distributed within the aluminum matrix and occasionally near the Al/ $B_4C$  interfaces.

Higher magnification SEM images of the areas marked B and C in Fig. 1c are presented in Fig. 1d, e. The white arrows indicate Si-rich particles, while P1, P2, and P3 indicate the locations selected for EDS spot analyses. Point P1 corresponds to the aluminum matrix, yielding a composition dominated by Al (~88.5 wt.%). Points P2 and P3 correspond to Si-rich particles, where silicon was identified as the major element, while aluminum was detected in lower amounts. The presence of aluminum in these spectra is attributed to beam interaction with the surrounding matrix due to the interaction volume exceeding the particle size. No iron was detected in these particles. The presence of these Si-containing phases is probably associated with residual impurities introduced during aluminum recycling or minor contamination during composite fabrication [28].

Although the composite is nominally composed of aluminum and  $B_4C$ , additional phases were detected by SEM and EDS, particularly the  $TiB_2$  interfacial layer and Si-rich particles. These features, however, cannot be clearly distinguished in the optical micrographs (Fig. 1a, b because of the limited reflectivity contrast between them and the aluminum matrix. This limitation highlights the relevance of the color-etching technique illustrated in Fig. 2.

In bright-field illumination (Fig. 2a), the microstructure appears similar to that observed in Fig. 1, indicating that the surface resembles a merely polished condition, with limited phase contrast. Under dark-field illumination (Fig. 2b), several particles and interfacial regions appear bright, indicating variations in surface reflectivity and providing preliminary evidence of microstructural heterogeneity. When polarized light is employed (Fig. 2c, d), significant color changes become evident as the analyzer angle is varied, enhancing phase contrast. Under these conditions, clear color differences are observed between

the aluminum matrix,  $B_4C$  particles, and bluish regions surrounding some particles. The same regions labeled A, B, and C are indicated in all images to facilitate direct comparison of microstructural features under the different illumination conditions.

The same region analyzed by optical microscopy in Fig. 2 was subsequently examined by SEM and EDS, as shown in Fig. 3.

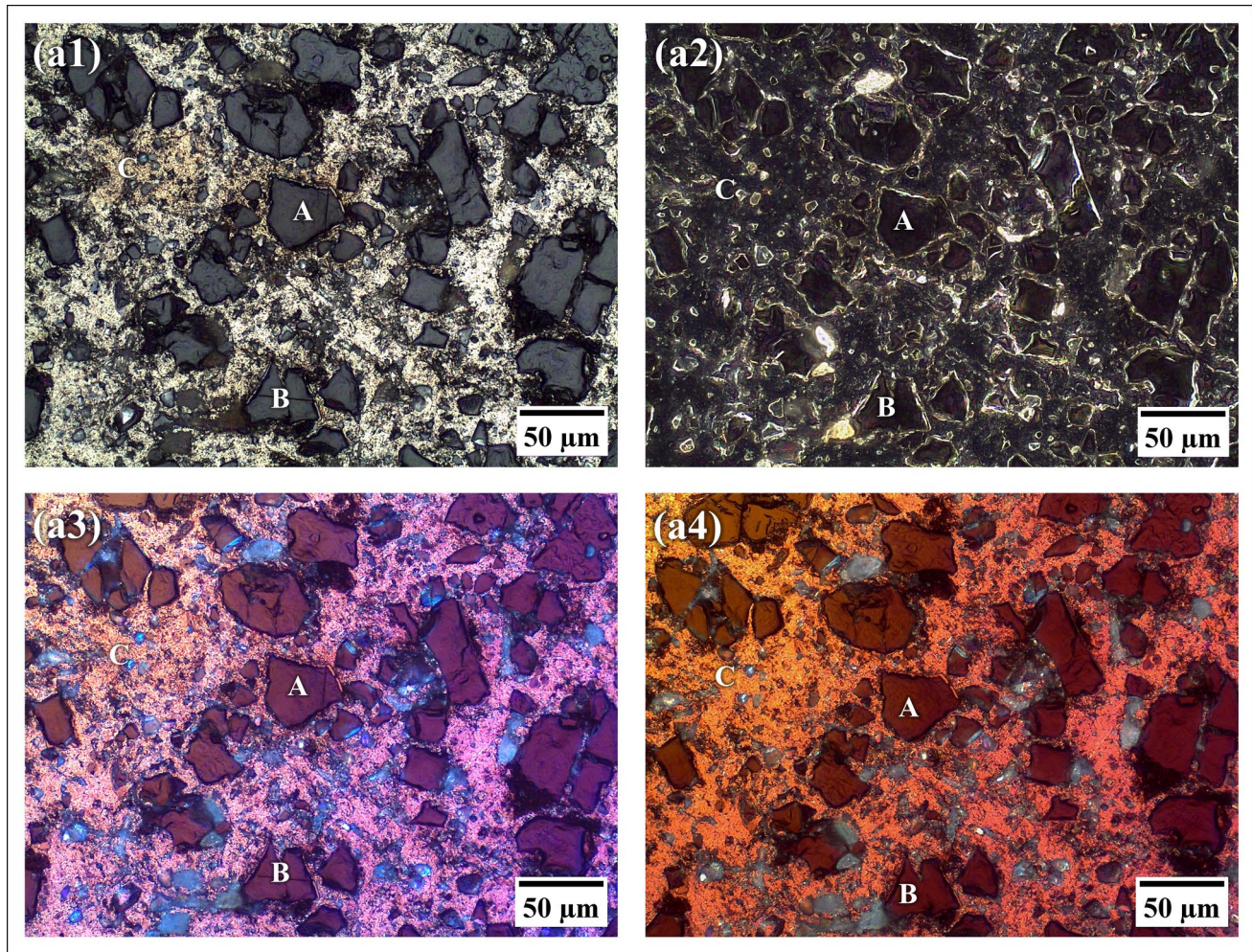
Regions A, B, and C indicated in Fig. 3a correspond to the same locations previously identified in Fig. 2, allowing direct correlation between optical contrast and compositional variations.

Figure 3a1 presents the EDS spectrum obtained from the analyzed area together with the corresponding semi-quantitative chemical composition, indicating B (65.9 wt.%), C (13.6 wt.%), O (4.7 wt.%), F (0.02 wt.%), Al (12.3 wt.%), Si (1.5 wt.%), Ti (1.9 wt.%), and Fe (0.04 wt.%). However, the contents of boron and carbon must be interpreted cautiously since they correspond to light elements and their quantification by EDS is inherently uncertain due to low X-ray yield and absorption effects. Likewise, Fe and F contents are extremely low and approach the detection limit of the technique. The presence of fluorine is attributed to residual effects from sample preparation using fluoroboric acid during color etching rather than representing a constituent phase of the composite. In addition, fluorine-containing reaction products formed during etching are typically highly soluble, which prevents their stable retention on the surface and explains the very low residual signal detected [34].

Elemental distribution maps further clarify phase distribution. The Al map in Fig. 3a2 clearly reveals the aluminum matrix, while  $B_4C$  particles appear dark due to the absence of aluminum signal. The Ti map in Fig. 3a3 shows enrichment surrounding  $B_4C$  particles, confirming the formation of a  $TiB_2$  interfacial layer produced by interfacial reactions during composite processing.

The Si map in Fig. 3a4 reveals discrete Si-rich particles dispersed within the matrix. Importantly, no iron enrichment is observed in these particles, indicating that they are not Al–Fe–Si intermetallic compounds but correspond instead to silicon-rich precipitates. This observation is consistent with aluminum metallurgy, where Fe has very limited solubility in Al and typically precipitates as Fe-containing intermetallic phases. Furthermore, according to the Al–Si phase diagram, when silicon exceeds its solubility limit in aluminum, Si-rich particles precipitate, explaining their presence in the matrix [35].

The oxygen map (Fig. 3a5) shows increased signal near some  $B_4C$ /matrix interfaces, suggesting localized oxidation of the aluminum matrix. This behavior is consistent with localized electrochemical interactions in which  $B_4C$  particles may promote localized anodic dissolution and subsequent oxide formation in adjacent aluminum regions.



**Fig. 2** Optical micrographs of the color-etched AA1100–25 vol.% B<sub>4</sub>C composite obtained using different observation modes: **a** bright field; **b** dark field; under polarized light with analyzer angles of **c** 65°

and **d** 55°. Regions A, B, and C correspond to the same locations highlighted in Figs. 3 and 4.

Finally, Fe and F maps (Fig. 3a6, a7) show weak and spatially dispersed signals. The fluorine signal is attributed to residual contributions from the electrolytic etching process, and fluorine-containing species formed during etching are highly soluble, preventing their stable retention on the surface. In contrast, although iron is present in the alloy, no Fe enrichment was detected in the analyzed region, indicating that this element is not associated with specific microstructural features in this area and occurs only as a trace background contribution.

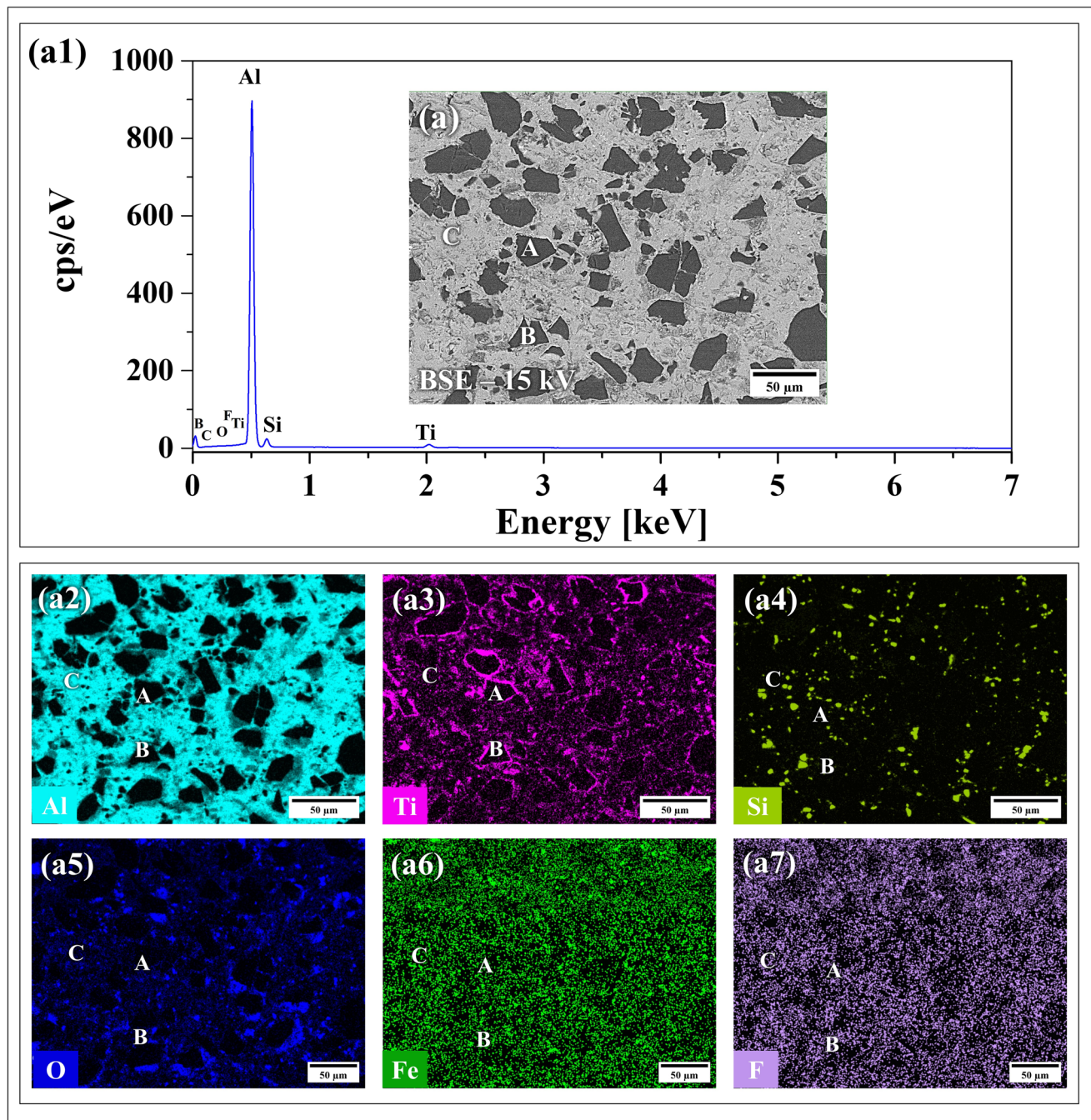
Figure 4 presents higher magnification SEM imaging and compositional analysis of the region shown in Fig. 2c, enabling a more detailed microstructural assessment.

In the optical micrograph (Fig. 4a), the surface exhibits distinct color regions generated by the chemical etching. B<sub>4</sub>C particles appear dark brown, the aluminum matrix displays a pinkish tone, and several bright blue areas are distributed throughout the matrix. These color contrasts arise

from differences in composition and local oxidation behavior among the constituent phases.

The corresponding EDS elemental map as shown in Fig. 4b, displayed in palette mode, represents the relative signal intensity distribution, with aluminum mainly represented by blue tones, silicon appearing as bright white regions, and other elements displayed in green and red according to their relative intensities. Since this representation is qualitative, it reflects regions where certain elements are abundant rather than providing quantitative composition. In this map, B<sub>4</sub>C particles appear as areas depleted in aluminum signal, while regions surrounding some particles exhibit compositional variations associated with interfacial reaction products.

The blue arrows in Fig. 4a indicate a thin, continuous border surrounding the B<sub>4</sub>C particles, attributed to the formation of a TiB<sub>2</sub> interfacial layer. Under optical illumination, this layer appears slightly darker than the aluminum

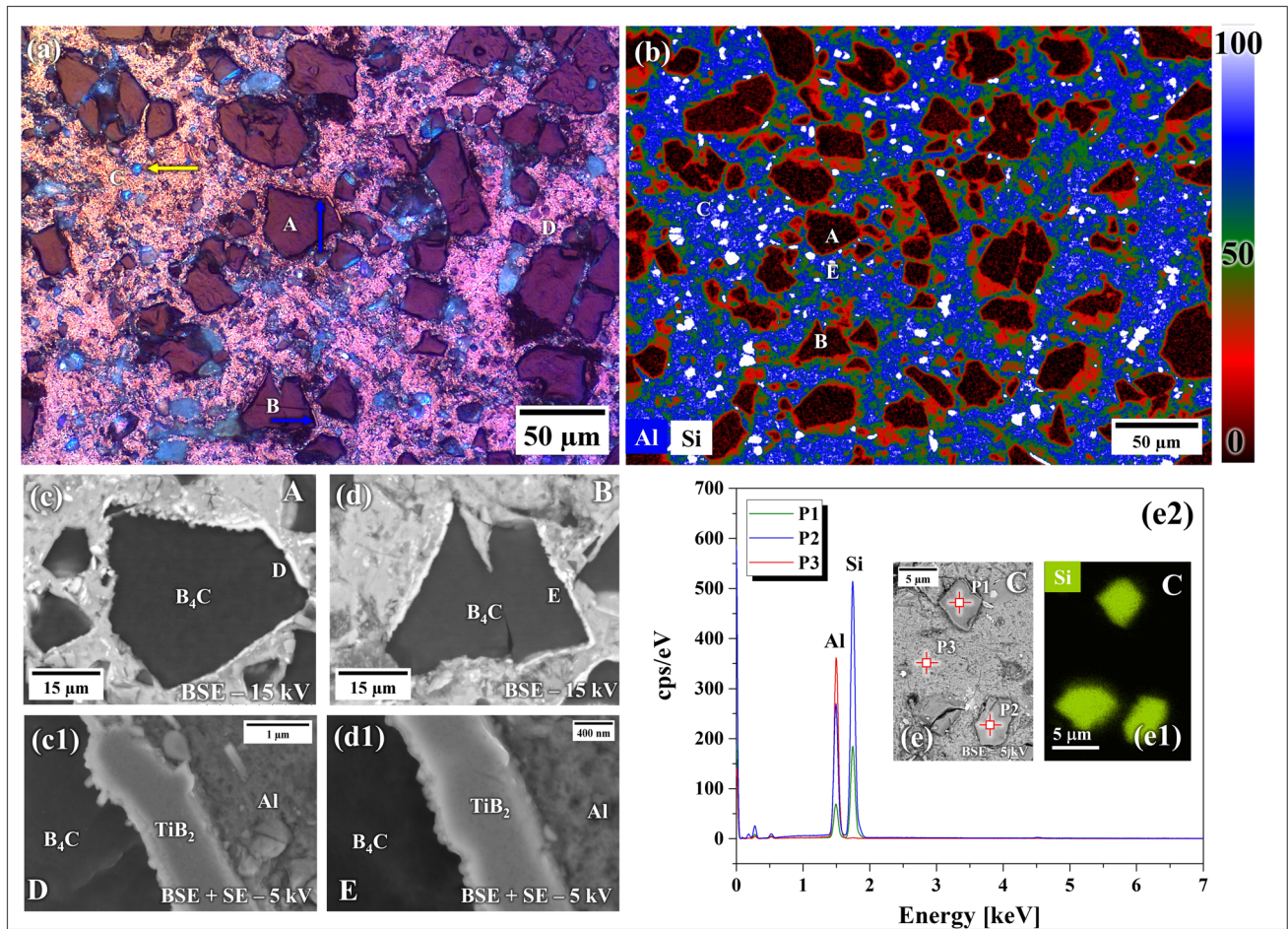


**Fig. 3** **a1** Scanning electron micrograph of the same region previously shown in Fig. 2, together with the corresponding EDS spectrum and elemental maps obtained from this region: **a2** Al K $\alpha$ , **a3** Ti K $\alpha$ , **a4** Si K $\alpha$ , **a5** O K $\alpha$ , **a6** Fe K $\alpha$ , and **a7** F K $\alpha$  distributions. Regions

B and C correspond to the same locations highlighted in Figs. 2 and 4. All analyses were performed at an accelerating voltage of 10 kV using backscattered electron detection (BSE).

matrix but brighter than the adjacent B<sub>4</sub>C phase, reflecting its intermediate optical response due to differences in reflectivity and surface oxidation. The yellow arrow in Fig. 4a points to bright blue features associated with Si-containing particles dispersed within the matrix.

The B<sub>4</sub>C particles labeled A and B in Fig. 4a correspond to the regions examined by SEM in Fig. 4c, d. In the backscattered electron images, the B<sub>4</sub>C reinforcement appears darker than the matrix, whereas the surrounding TiB<sub>2</sub> interfacial film appears brighter due to the higher average



**Fig. 4** **a** Optical micrograph of the color-etched AA1100–25 vol.% B<sub>4</sub>C composite, showing the distribution of B<sub>4</sub>C particles (dark brown), aluminum matrix (pinkish tone), and Si-rich phases (blue); **b** EDS elemental map in palette mode corresponding to the region shown in **a**; **c** and **d** Scanning electron micrographs of B<sub>4</sub>C particles corresponding to regions A and B indicated in **a**; **c1** and **d1** higher magnification images of regions D and E marked in **c** and **d**, respectively, acquired using simultaneous backscattered and secondary elec-

tron signals, showing the TiB<sub>2</sub> layer formed at the B<sub>4</sub>C/Al interface; **e** scanning electron micrograph corresponding to region C indicated in **a**; **e1** EDS elemental map obtained from the region shown in **e**; **e2** EDS spectra corresponding to the spot analyses performed at points P1, P2, and P3 indicated in **e**; SEM and EDS analyses were conducted at accelerating voltages of 15 kV (BSE imaging and EDS acquisition) and 5 kV for simultaneous BSE and SE imaging.

atomic number of titanium compared with aluminum and boron [33]. Higher magnification views of regions D and E, indicated in Fig. 4c, d, are presented in Fig. 4c1, d1, revealing detailed features of the B<sub>4</sub>C/TiB<sub>2</sub>/Al interfaces. Images acquired using simultaneous BSE and SE signals show the continuity and relatively uniform thickness of the TiB<sub>2</sub> layer, confirming its formation during processing and its intimate bonding with both the aluminum matrix and the B<sub>4</sub>C particles [22].

The association between titanium and these interfacial regions is also supported by the Ti elemental map presented in Fig. 3a3, which shows Ti enrichment along particle boundaries corresponding to regions D and E. This elemental distribution confirms that Ti is concentrated at the interface between B<sub>4</sub>C particles and the aluminum

matrix, consistent with the formation of a TiB<sub>2</sub> reaction layer responsible for the optical contrast observed around the reinforcement. Boron detection at this interface is limited due to the low atomic number of the element [33] and the small thickness of the interfacial layer, which reduces EDS sensitivity in this region.

Figure 4e presents a higher magnification SEM image of region C indicated in Fig. 4a, while Fig. 4e1 shows the corresponding EDS map confirming that this region is enriched in silicon. EDS analyses performed at points P1, P2, and P3 (Fig. 4e2) further clarify local compositions. Point P3 corresponds to the aluminum matrix and is dominated by aluminum signal, whereas points P1 and P2 correspond to Si-rich particles. The measured silicon content reaches approximately 25 wt.% at P1 and increases to about 88 wt.%

at P2, which is reflected by the stronger Si peak in the corresponding EDS spectra. Aluminum is still detected at P1 and P2; however, this contribution is attributed to beam interaction with the surrounding aluminum due to the electron interaction volume exceeding the particle size. This interpretation is supported by the palette map in Fig. 4b, where region C shows strong silicon signal associated with reduced aluminum intensity, confirming that these particles correspond to Si-rich phases rather than matrix regions.

The different colors observed in Figs. 2b, d and 4a result from variations in oxidation kinetics and oxide-film thickness across the constituent phases during the etching process. The ceramic  $B_4C$  particles are chemically inert and dissolve more slowly, retaining a dark brown appearance. The aluminum matrix, being more reactive, develops an oxide film of intermediate thickness that reflects a pinkish hue. The  $TiB_2$  interfacial layer exhibits a slightly different oxidation behaviors, producing subtle brown contours around the reinforcement, while the Si-rich inclusions exhibit a blue reflection. These optical responses arise from thin-film interference effects, in which local differences in oxide thickness and refractive index modify light absorption and reflection, generating the characteristic color contrast. These color differences are therefore controlled not only by composition but also by local electrochemical reactivity during anodic etching.

Therefore, the color-etching technique employed in this study effectively distinguishes the constituent phases of the composite and enables the visual identification of  $TiB_2$  interfacial layer and Si-rich particles, which are otherwise difficult to identify in polished specimens. This approach facilitates microstructural evaluation and quality control of Al- $B_4C$  composites and demonstrates that color-etching strategies can be successfully designed for aluminum-based composites. However, their application to systems reinforced with other ceramic particles requires specific experimental validation, since different reinforcements may respond differently to electrolytic attack.

## Conclusion

The color-etching methodology proposed in this study proved effective in revealing the microstructural constituents of the AA1100–25 vol.%  $B_4C$  composite. It enabled clear optical differentiation of the aluminum matrix,  $B_4C$  reinforcement, the  $TiB_2$  interfacial layer, and Si-rich inclusions, which are otherwise difficult to identify in polished or unetched specimens. The color contrast originates from the distinct oxidation behavior and oxide-film thickness developed on each constituent during etching, thereby providing a reliable approach for phase identification under reflected light. This methodology provides a practical complement to

electron microscopy and can be applied for microstructural evaluation and quality control of aluminum-based ceramic composites.

**Acknowledgements** The authors acknowledge the São Paulo Research Foundation (FAPESP) for supporting João Victor de Sousa Araujo (Proc. 24/01015-6) and Nathanael W. Morais (25/12987-1).

**Author contributions** João Victor de S. Araujo involved in investigation, visualization, and writing—original draft. Nathanael W. Morais took part in investigation. Isolda Costa involved supervision.

**Funding** The Article Processing Charge (APC) for the publication of this research was funded by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) (ROR identifier: 00x0ma614).

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