Technical Sciences
Academy of Romania
www.jesi.astr.ro

Journal of Engineering Sciences and Innovation
Volume 9, Issue 2 / 2024, pp. 129 - 140

C. Chemical Engineering, Materials Science and Engineering

Received ***10 February 2024*** *Accepted* ***14 June 2024***
Received in revised form ***24 Apryl 2024***

Advanced fabrication and characterization of ZrB_2 reinforcement AA6063 matrix composites

PETRU MOLDOVAN[1], MIHAI BUȚU[1*], MIHAI BRÂNZEI, CONSTANTIN-DOMENIC STĂNCEL[1], LUCIAN ROȘU[1]

[1]*National University of Science and Technology POLITEHNICA Bucharest, Splaiul Independentei, 313, Bucharest, Romania*
[2] *COMOTI - Romanian Research & Development Institute for Gas Turbines, B-dul Iuliu Maniu, 220D, sector 6, 061126, Bucharest, Romania.*

Abstract. The development of aluminum matrix composites (AMCs) reinforced with ZrB2 particles represents a significant advancement in materials engineering, combining lightweight properties with exceptional mechanical. This paper presents a comprehensive study on the in-situ fabrication of AA6063/ZrB2 composites, exploring the material properties, and potential industrial applications. By employing in-situ reaction techniques, ZrB2 particles are synthesized directly within an aluminum alloy matrix, resulting in a strong interfacial bond and uniform dispersion. The effects of fabrication parameters on the microstructure and properties of the composites are thoroughly investigated. The study highlights the improvements in hardness positioning these composites as promising materials for aerospace, automotive, and high-temperature applications.

Keywords: MMCs, ZrB_2 particles, AA6063/ZrB_2 composites.

1. Introduction

Metal Matrix Composites (MMCs) have emerged as a type of material that use the interaction of metal matrices and reinforcing phases to achieve properties that neither component could achieve on its own. Aluminium matrix composites (AMCs) have gained popularity due to their lightweight, high strength, and superior thermal properties, making them ideal for aerospace, automotive, and high-temperature applications [1]. The incorporation of ceramic particles such as zirconium diboride (ZrB_2) into aluminium matrix is a promising method for

* Correspondence address: stancel.constantin@yahoo.com

improving the mechanical strength, wear resistance, and thermal stability of these composites [2].

ZrB_2 is distinguished by its high melting point, exceptional hardness, and excellent thermal conductivity, which, when combined with aluminium, results in a composite material that exhibits enhanced performance under extreme conditions [3-4]. The fabrication of AMCs reinforced with ZrB_2 particles, however, presents challenges, particularly regarding the uniform dispersion of ceramic particles within the metal matrix and the achievement of strong interfacial bonding between the two phases. Addressing these challenges is crucial for optimizing the mechanical and thermal properties of the composites [3-4].

Recent advancements in fabrication techniques, especially in-situ synthesis methods, have shown promise in overcoming these obstacles. The in-situ formation of ZrB_2 particles within an aluminium alloy matrix not only ensures a uniform distribution of the reinforcing phase but also enhances the interfacial bonding, leading to composites with superior properties [5]. This study focuses on the in-situ fabrication of AA6063/ZrB_2 composites, exploring the thermodynamics of the process and the impact of various fabrication parameters on the microstructure and properties of the final composite material [5].

The objectives of this study are to develop a comprehensive understanding of the in-situ fabrication process for AA6063/ZrB_2 composites, to characterize the microstructural features and mechanical properties of the resulting materials, and to assess their potential applications in industries requiring materials with high strength-to-weight ratios and thermal stability. By addressing the gaps in current research and exploring novel approaches to composite fabrication, this study aims to contribute to the advancement of materials science and engineering, particularly in the development of high-performance AMCs suitable for demanding engineering applications [6].

2. Literature review

The exploration and development of Metal Matrix Composites (MMCs), especially those reinforced with ceramic particles, have been the subject of extensive research over the past few decades. Among the wide range of ceramics utilized, zirconium diboride (ZrB_2) stands out due to its exceptional properties, including high melting point, extreme hardness, and excellent thermal conductivity [7]. This section reviews the literature on MMCs, with a particular focus on aluminium matrix composites (AMCs) reinforced with ZrB_2 particles, highlighting the methodologies adopted for their fabrication, the resulting material properties, and their potential applications.

2.1. Fabrication techniques

The incorporation of ZrB_2 particles into aluminium matrices has been investigated through various techniques, with in-situ synthesis methods receiving particular attention for their ability to ensure a uniform dispersion and strong interfacial bonding between the matrix and the reinforcement [7]. Traditional methods like stir casting and powder metallurgy have been employed for the fabrication of ZrB_2 reinforced AMCs; however, they often result in particle agglomeration and weak interface bonding, limiting the mechanical properties of the composites [7]. In contrast, in-situ techniques involve the formation of ZrB_2 particles directly within the molten aluminium alloy, facilitating a cleaner and more robust interface and a more homogeneous distribution of the reinforcing phase. Studies have shown that parameters such as reaction temperature, holding time, and the nature of reactants significantly influence the size, morphology, and distribution of ZrB_2 particles within the aluminium matrix [8].

2.2. Material properties

The mechanical properties of AMCs reinforced with ZrB_2, such as hardness, tensile strength, and wear resistance, have been reported to improve significantly compared to unreinforced alloys [8]. The presence of hard ZrB_2 particles within the ductile aluminium matrix imparts a remarkable increase in composite hardness and strength through mechanisms like load transfer and Orowan strengthening. The thermal properties of these composites, including thermal conductivity and coefficient of thermal expansion, are also influenced by the ZrB_2 reinforcement, making them suitable for applications requiring high thermal stability [8].

2.3. Applications

The enhanced properties of ZrB_2 reinforced AMCs have opened new possibilities for their application in various demanding fields. In the aerospace industry, these composites are considered potential materials for structural components and heat shields, owing to their low density, high specific strength, and excellent thermal stability [9]. The automotive sector also shows great interest in these composites for components like brake rotors and engine parts, where improved wear resistance and thermal properties can significantly enhance performance and durability [10].

2.4. Challenges and future directions

Despite the promising properties and applications of ZrB_2 reinforced AMCs, challenges remain in optimizing the fabrication process for large-scale production and in further improving the material properties. Future research is directed towards refining in-situ synthesis techniques to control the microstructure more precisely, exploring the effects of different matrix alloys and reinforcement morphologies, and assessing the long-term performance of these composites in real-world applications [10].

3. Methodology

The methodology section of this article outlines the experimental procedures and analytical techniques employed in the fabrication and characterization of AA6063/ZrB_2 composites. The study emphasizes the in-situ reaction process for synthesizing ZrB_2 particles within the aluminium matrix, aiming for a uniform dispersion and strong interfacial bonding between the matrix and reinforcement [11]. The experimental setup, material selection, fabrication parameters, and characterization methods are detailed as follows:

3.1. Material selection

Matrix Material: Aluminium alloy AA6063 was selected as the matrix material due to its excellent formability, corrosion resistance, and suitability for extrusion processes. The alloy composition and pre-treatment conditions were standardized to ensure reproducibility [12].
Reinforcing Phase: Zirconium diboride (ZrB_2) particles were synthesized in-situ from a mixture of zirconium (K_2ZrF_6) and boron (KBF_4) sources. The purity and particle size of the starting materials were carefully controlled to influence the morphology and distribution of the reinforcing phase.

3.2. In-situ synthesis process

For the preparation of the reaction mixture, a meticulously calculated stoichiometric blend of zirconium and boron sources was composed. This blend was subsequently introduced into the molten AA6063 alloy, with measures taken to maintain a controlled atmosphere, thereby averting oxidation and contamination [13].
Regarding the reaction conditions, the temperature of the molten alloy was carefully regulated to remain above the melting point of AA6063 yet below the decomposition temperatures of the reactants. This was done to support the in-situ generation of ZrB_2 particles at a specific temperature of 850°C. The reaction parameters, including a duration of 2 minutes, a temperature setting of 850°C, and a stirring speed of 300 RPM, were optimized based on initial experiments. Such optimization was aimed at ensuring a uniform dispersion of ZrB_2 particles within the alloy.

3.3. Composite characterization

The microstructure of the fabricated composites was examined using optical microscopy (OM) and scanning electron microscopy (SEM), focusing on the distribution, morphology, and interfacial integrity of ZrB_2 particles within the AA6063 matrix. Energy-dispersive X-ray spectroscopy (EDS) was employed to analyse the elemental composition at the micro-level.

Mechanical testing, including hardness measurements, tensile testing, and compressive strength tests, was conducted to evaluate the effect of ZrB_2 reinforcement on the mechanical properties of the composites. Standard ASTM procedures were followed to ensure accuracy and repeatability.

4. Results and discussion

The fabrication of AA6063/ZrB_2 composites via an in-situ reaction process led to the successful synthesis of ZrB_2 particles within the aluminium matrix. The results obtained from microstructural analysis, mechanical testing are discussed in this section, revealing the significant impact of ZrB_2 reinforcement on the composite's properties.

4.1. Microstructural analysis

For OM the samples were etched with Keller’s reagent and were analysed at different magnifications (Fig. 4.1, Fig. 4.2. Fig. 4.3, Fig. 4.4) marked with the letters A B C D, for 2.5, 5, 7.5 and 10% ZrB_2 respectively.

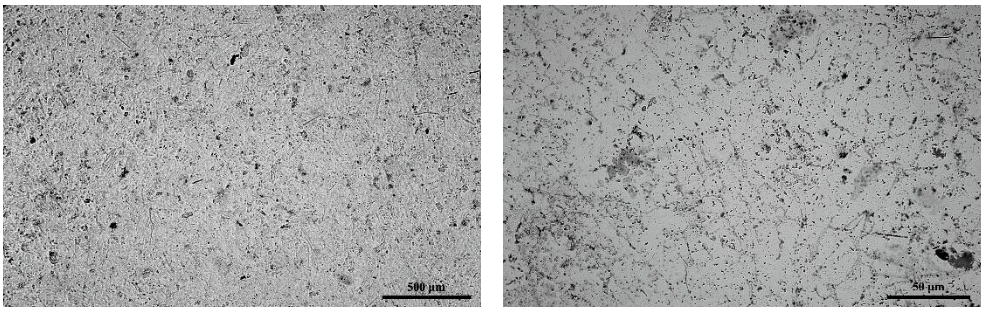

Fig. 4.1. The microstructure of the etched A (2.5% ZrB_2) composite, at different magnifications.

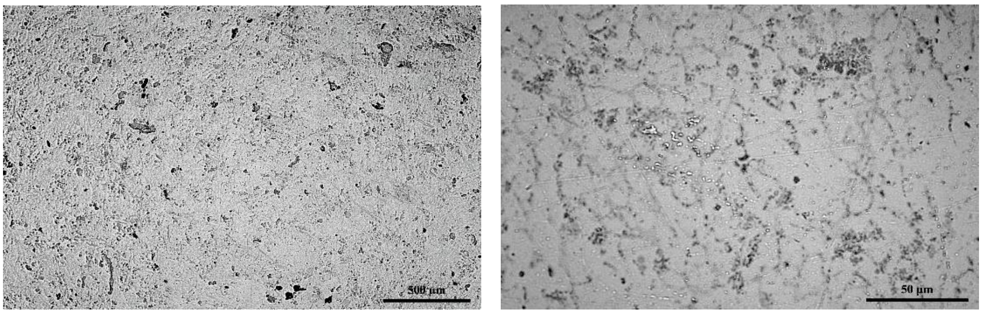

Fig. 4.2. The microstructure of the etched B (5% ZrB2) composite, at different magnifications.

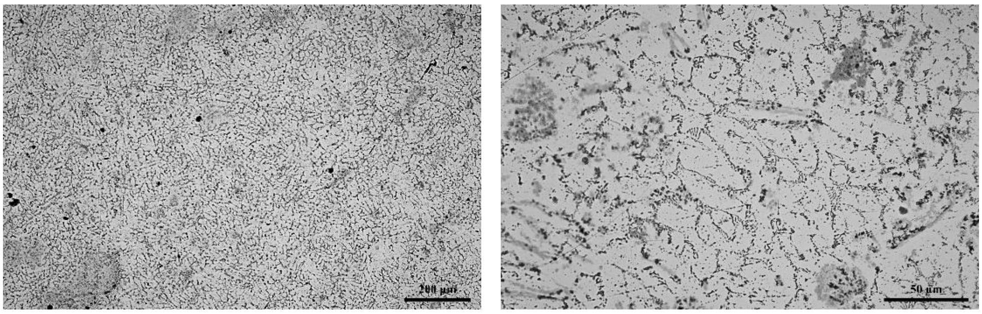

Fig. 4.3. The microstructure of the etched C (7.5% ZrB_2) composite, at different magnifications.

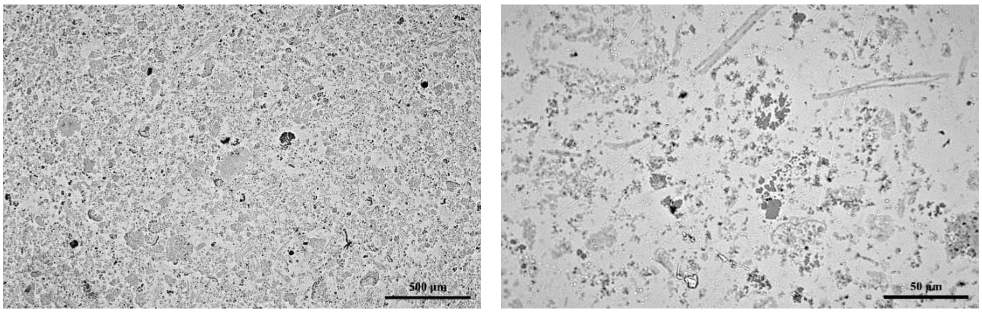

Fig. 4.4. The microstructure of the etched C (10.0% ZrB_2) composite, at different magnifications.

The optical micrographs of composites exhibit the nearly uniform distribution of ZrB_2 particles. The micrographs of composites reinforced with increasing of ZrB_2 particles shown in Fig. 4.2 – 4.4 clearly indicate that the grain size of matrix phase refines on in-situ formation of ZrB_2 particles due to restriction in the movement of solidification front.

Scanning electron microscope (SEM) was used to study morphology and distribution of ZrB_2 phase in the composites (Fig. 4.5, Fig. 4.6, Fig. 4.7, Fig. 4.8).

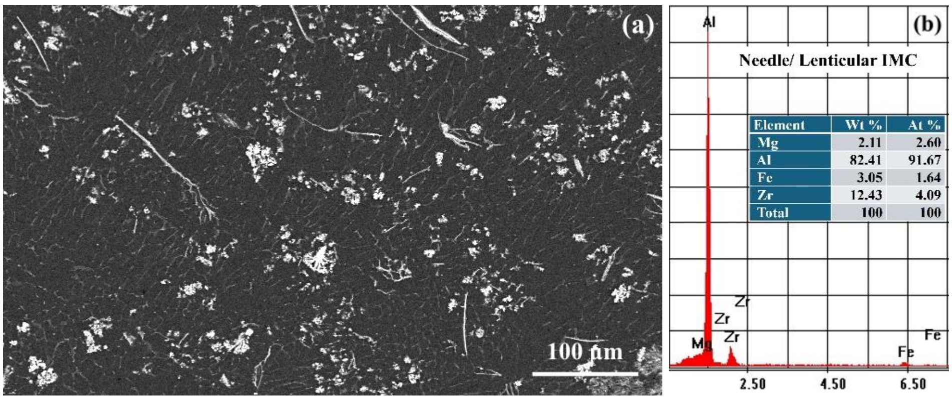

Fig. 4.5. SEM image (a) and EDS analysis of AA6063/ZrB_2 IMC (b) with 2.5% ZrB_2.

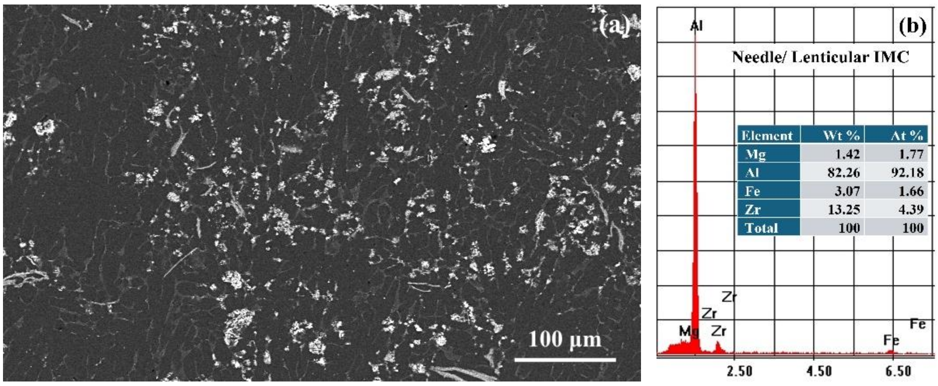

Fig. 4.6. SEM image (a) and EDS analysis of AA6063/ZrB_2 IMC (b) with 5.0% ZrB_2

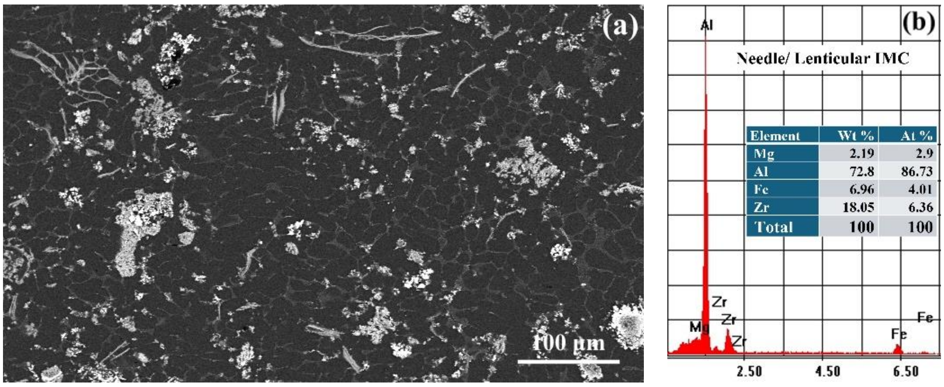

Fig. SEM image (a) and EDS analysis of AA6063/ZrB_2 IMC (b) with 7.5% ZrB_2

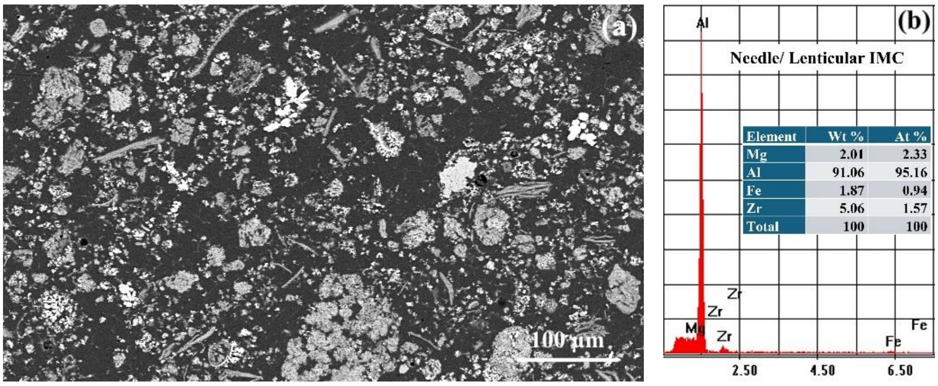

Fig. 4.8. SEM image (a) and EDS analysis of AA6063/ZrB_2 IMC (b) with 10.0% ZrB_2.

It is observed that in-situ formed ZrB_2 particles are uniformly distributed in the Al matrix. Agglomeration of ZrB_2 particles is also seen and that increases with the increase in the amount of ZrB_2 particles. Density difference between the matrix (AA6063) and ZrB_2 particles is more than 2 g/cm^3, hence the ZrB_2 particle can suspend for a long time in the melt which is required to obtain the uniform distribution of ZrB_2 particles in the composites. Presence of clear interface may be attributed to thermodynamic stability of ZrB_2 particles.

4.2. XRD analysis and in-situ formation of ZrB_2 particles

In Fig. 4.9 – 4.12 are shown XRD patterns of AA6063/ZrB_2 composites.
The diffraction profiles obtained with the PANalytical X'Pert PRO diffractometer provide detailed insights into the structural properties of the AA 6063 aluminium alloy, which has been in-situ reinforced with Zirconium Diboride (ZrB_2) at concentrations of 2.5%, 5%, 7.5%, and 10% respectively. These profiles are important in determining the phase composition and degree of crystallinity of composite materials, as well as demonstrating how the ZrB_2 reinforcing phase affects the alloy's microstructural properties.

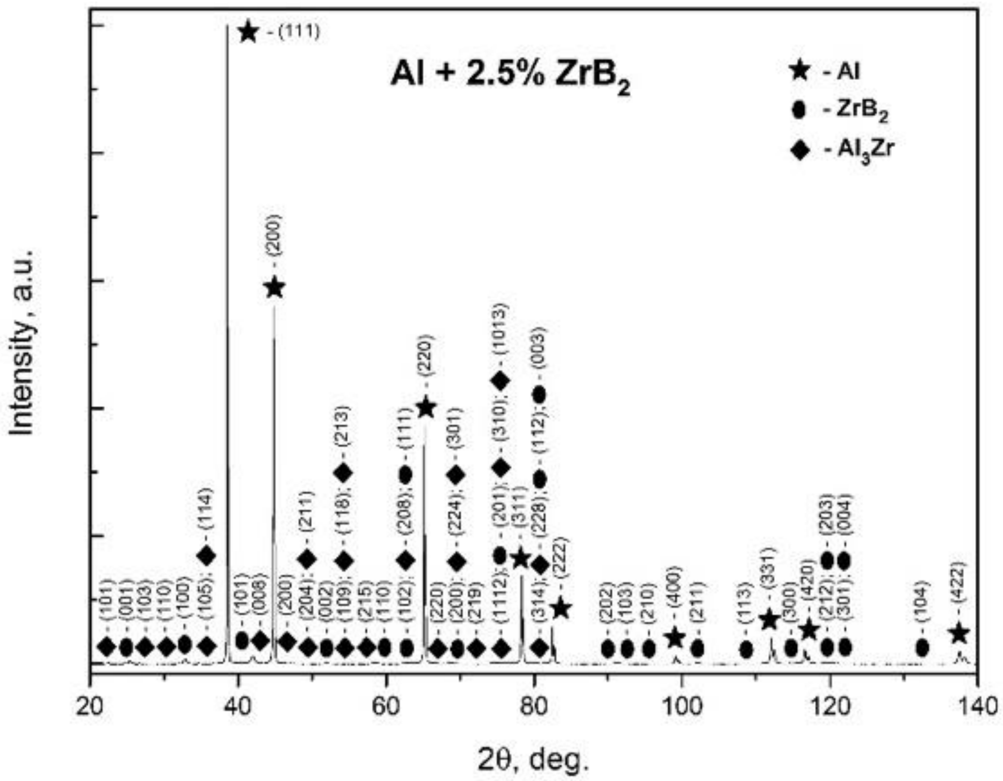

Fig. 4.9. XRD patterns of AA 6063/2.5% ZrB_2 composites after 10 min of reaction time.

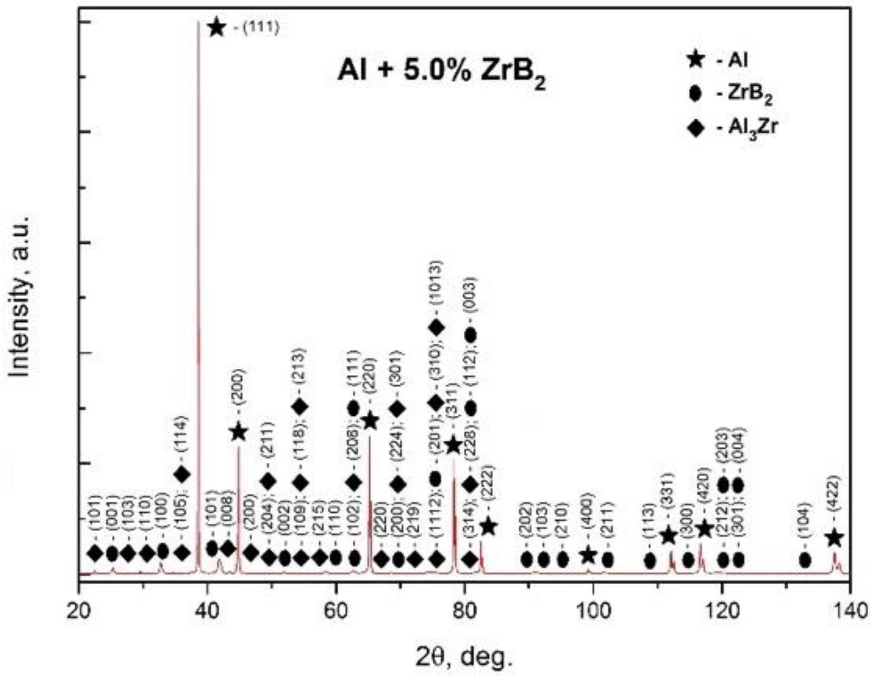

Fig. 4.10. XRD patterns of AA 6063/5% ZrB_2 composites after 10 min of reaction time.

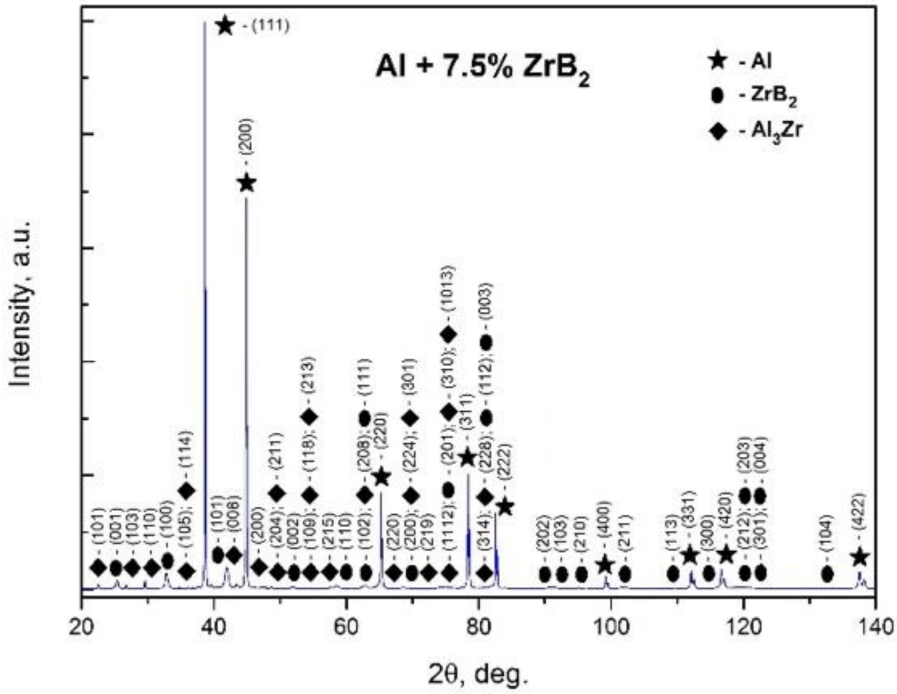

Fig. 4.11. XRD patterns of AA 6063/7.5% ZrB_2 composites after 10 min of reaction time.

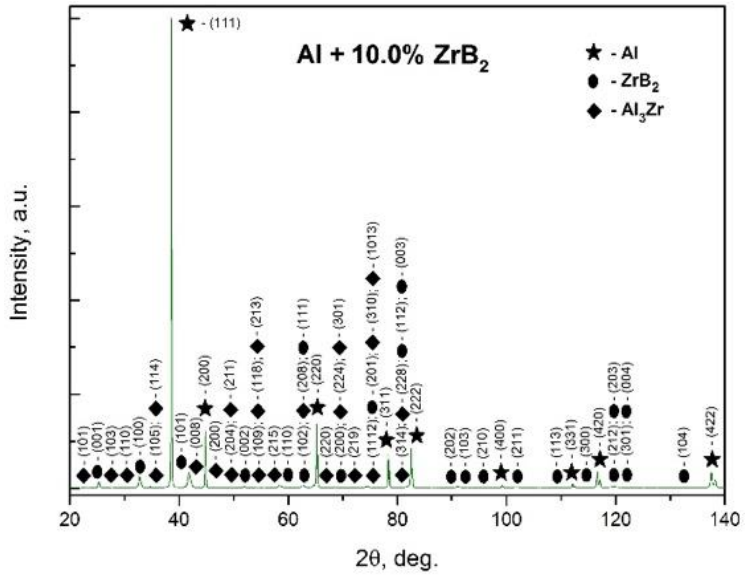

Fig. 4.12. XRD patterns of AA 6063/10% ZrB_2 composites after 10 min of reaction time.

If the reaction time is 10 min it is more likely to form Al_3Zr. After 35 min of reaction time was given for complete transformations of Al_3Zr to ZrB_2. Peaks of Al_3Zr are not observed.

4.3. Microhardness HV

Table 4.1 shows the variation of Vickers hardness by using a Wilson VH1102 Micro Hardness Testers.

Table 4.1. Microhardness HV AA 6063/ZrB_2 for samples A, B, C and D 25gf (20s)

A (HV)	B (HV)	C (HV)	D (HV)
109	137	169	185
113	408	169	194
137	137	177	194
137	194	162	225
214	169	214	225
132	137	162	214
185	214	132	204
185	214	250	165
169	225	194	335
177			204
HV Mean			
155,8	203,889	181	214,5

4.4. Discussion

The uniform dispersion of ZrB_2 particles and the strong interfacial bonding achieved through the in-situ reaction process are key factors contributing to the enhanced mechanical and thermal properties of the composites. The methodology employed in this study effectively addresses common challenges in composite fabrication, such as particle agglomeration and weak interfacial bonding, which are critical for optimizing the performance of MMCs. The findings underscore the significance of processing conditions on the properties of metal matrix composites and open avenues for further research into in-situ fabrication techniques.
The results of this study not only provide insights into the development of high-performance AA6063/ZrB_2 composites but also demonstrate the potential of in-situ synthesis methods for the fabrication of MMCs with tailored properties for specific applications.

5. Conclusions

The work presented has led to the development of new materials The following results can be considered original:
- A comprehensive documentary study on composite materials, classifying them by base matrix and reinforcing elements.
- A documentary study on the structure and properties of composite materials in comparison with the structure and properties of classic metallic materials.
- Microstructural characterization of AA6063/ZrB_2 composite materials through optical and electronic microscopy (SEM and EDS).
- Characterization of the in-situ obtained composites through X-ray diffraction (XRD).
- Vickers microhardness in different areas of the composite materials reinforced with ZrB_2 ceramic particles.

References

[1] Kainer K.U., *Metal matrix composites custom-made materials for automotive and aerospace engineering*, Basics of Metal Matrix Composites, Weinheim, WILEY-VCH Verlag GmbH & Co, 2006.
[2] Singh B., Lal H., Singh G., Singh P., *Effect on properties of aluminum by adding alumina slag as a strengthener*, International Journal of Mechanical Engineering and Robotics Research, **2**, 3, 2013, p. 96 - 100.
[3] *The Material and Process Requirements in Disc Brake Engineering Essay*, UKEssays, November 2018. [Online]. Available: https://www.ukessays.com/essays/engineering/the-material-and-process-requirements-in-disc-brake-engineering-essay.php.
[4] Sornakumar T., Marimuthu K., *Machining studies of die-cast aluminum alloy-silicon carbide composites*, International Journal of Minerals, Metallurgy and Materials, **17**, 2010, p. 648 - 653.
[5] Vinayagam M., R. K., Ravichandran M., *Synthesis, characterization, and properties of stir cast AA6351-aluminium nitride (AlN) composites*, Journal of Materials Research, **31**, 2016, p. 1-8.
[6] Chen X.-H., Yan H., *Fabrication of nanosized Al_2O_3 reinforced aluminum matrix composites by subtype multifrequency ultrasonic vibration*, Journal of Materials Research, **30**, 2015, p. 2197 - 2209.

[7] Mohanavel V., Kumar S.S., Sathish T., Anand K., *Effect of ZrB_2 content on mechanical and microstructural characterization of AA6063 aluminum matrix composites*, Materials Today: Proceedings, **5**, 5, 2018, p. 13601 - 13605.
[8] Cantor B., Dunne F., Stone I., *Metal and Ceramic Matrix Composites*, London: Institute of Physics Publishing, 2004.
[9] Sharma A.K., Bhandari R., Aherwar A., Rimašauskienė R., Pinca-Bretotean C., *A study of advancement in application opportunities of aluminum metal matrix composites*, Materials Today: Proceedings, **26**, 2, 2020, p. 2419 - 2424.
[10] Surappa M., *Aluminium matrix composites: challenges and opportunities*, Sadhana, **28**, 2003, p. 319 - 334.
[11] Callister Jr. W.D., Rethwisch D.G., Chapter 16. *Composites*, Materials Science and Engineering: An Introduction, Wiley, 2006, p. 577 - 617.
[12] Girot F.A., Majidi A.P., Chou T.W., *Metal Matrix Composites*, Encyclopedia of Physical Science and Technology (Third Edition), 2003, p. 485 - 493.
[13] Chawla N., Chawla K.K., *Metal Matrix Composites*, New York, Springer, 2013.
[14] Buțu Mihai, Moldovan Petru, Roșu Lucian, Stăncel Constantin, Ogica Cătălin, Buțu Larisa, Marinescu Marilena, *Research studies on studies on obtaining metastable intermetallic structures in 6xxx/borides composites*, U.P.B SCI. BULL. Series B-Chemistry and Materials Sciences, **82**, 2, 2020, p. 185 - 198.