

**THE EFFECT OF REACTANT COMPOSITIONS ON THE
MICROSTRUCTURE OF CERAMIC MATERIALS FORMED BY
COMBUSTION SYNTHESIS**

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ABSTRACT

Ceramics have been attractive as advance materials for high temperature applications due to their advantages such as high modulus, high hardness, high melting temperature, and high corrosion resistance. Of a broad range of ceramics, titanium carbide and aluminide have developed into one of the most interesting ceramics since they have high strength and good wear resistance at elevated ceramic materials. Producing TiC-Al₂O₃ from economical material (TiO₂) is less expensive than that from their elemental powders. The combustion synthesis (CS) process offers significant advantages to fabricate TiC-Al₂O₃ since it produces high exothermic reaction and takes a short processing time. The objective of this study was to explore the combustion synthesis of TiC-Al₂O₃ and investigate the effect of reactant compositions on the combustion process and microstructure of synthesized products. The combustion synthesis was carried out in a combustion chamber with an atmospheric condition. The ignition technique to initiate the CS was performed using an arc flame. The microstructure and mechanical properties of synthesized products were observed. The result of this research would benefit for producing a high performance ceramic material obtained from economical material with an efficient process.

Keywords: Ceramics, TiC-Al₂O₃, TiO₂, Combustion synthesis, Arc flame, Microstructure

1. INTRODUCTION

1.1 Background

Ceramic materials play a crucial role in modern industry thanks to their superior properties, such as high thermal stability, wear resistance, and exceptional hardness. These characteristics make ceramics highly desirable for various applications, including refractories, protective layers (armor), and cutting tools. The functional quality of ceramic materials is highly dependent on the microstructure formed during the synthesis process.

Conventionally, ceramic materials are often produced through the Powder Metallurgy technique, which involves mixing, compaction, and high-temperature sintering over a long duration. Although this method has proven effective, the process generally requires high energy consumption and a complex processing environment, thus prompting the need to develop more efficient and economical alternative synthesis methods.

1.2 Problem Formulation

Based on the background that has been described, this research formulates the following problems:

1. How do variations in reactant composition influence the combustion characteristics of TiC–Al₂O₃ ceramics synthesized via CS?
2. What is the relationship between the reactant composition and the microstructural properties (grain size, porosity, and phase distribution) of the synthesized products?
3. How can compositional control optimize the quality and performance of TiC–Al₂O₃ ceramics?

1.3 Research Limitation

Based on the existing problem formulation, this research concentrates on:

- a. Objek penelitian adalah sebuah ruangan dengan dimensi panjang 10,45 m, lebar 8,10 m, dan tinggi 3,93 m yang dilengkapi dengan dua unit AC tipe split.
- b. The study focuses only on the Ti–C–Al–O system, using TiO₂, Al, and C powders as starting materials.
- c. The synthesis process is performed under atmospheric conditions, initiated using an arc flame ignition technique.
- d. Variations in reactant compositions are limited to 0%, 20%, 40%, 60%, 80%, and 100% excess TiO₂ and C.
- e. Characterization is restricted to X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) for phase and microstructural analysis.
- f. The study does not include mechanical testing or thermodynamic modeling of the combustion process.

1.4 Objective of the Study

- a. To synthesize ceramic materials using the combustion synthesis (CS) process.
- b. To investigate the influence of reactant composition on the microstructure of the synthesized TiC–Al₂O₃ ceramics.

2. METHODOLOGY

This study aimed to produce ceramic materials using combustion synthesis (CS) and investigate the effect of reactant compositions on the process and the synthesized product's microstructure.

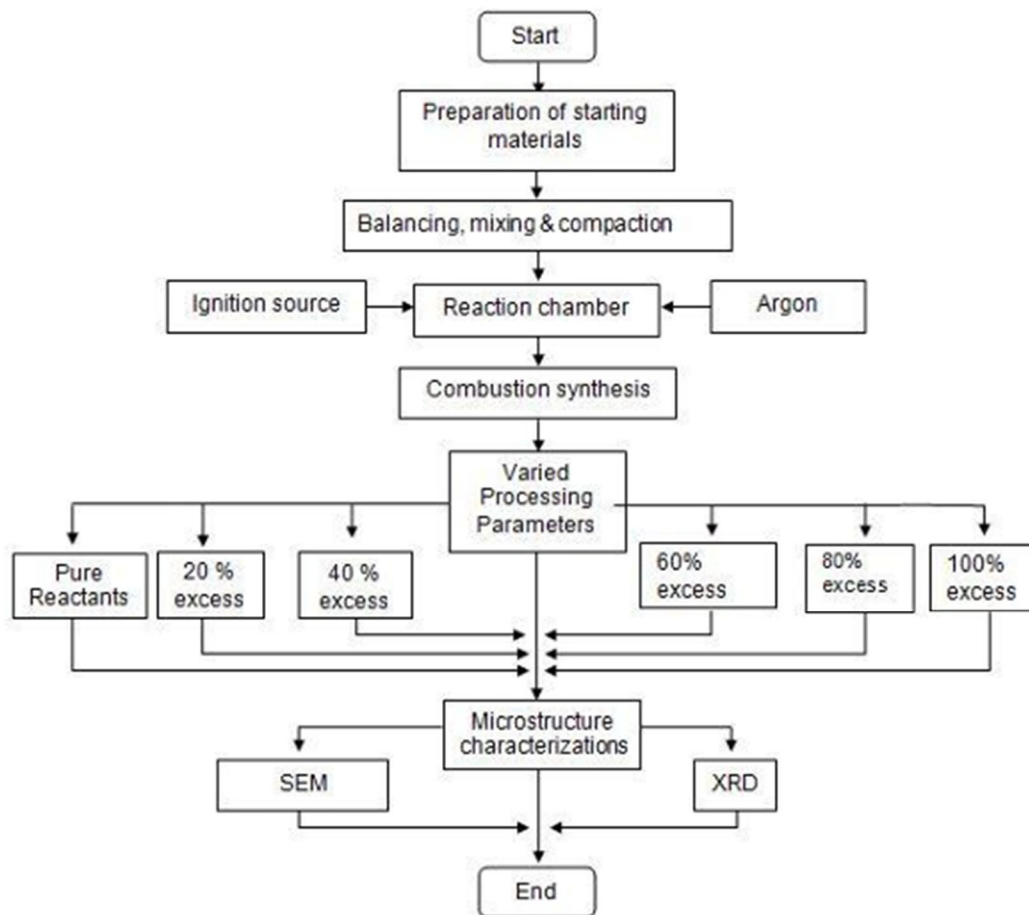


Figure 1. Flow Chart of project

2.1 Materials

The powders utilized as starting materials in this research were aluminum (Al), titanium oxide (TiO₂), and carbon black (C). Alumina (Al₂O₃) powder was also used. The focus was on producing the composite ceramic material TiC- Al₂O₃ from the more economical material, TiO₂, compared to using elemental powder.



Figure 2. TiO₂ Powder

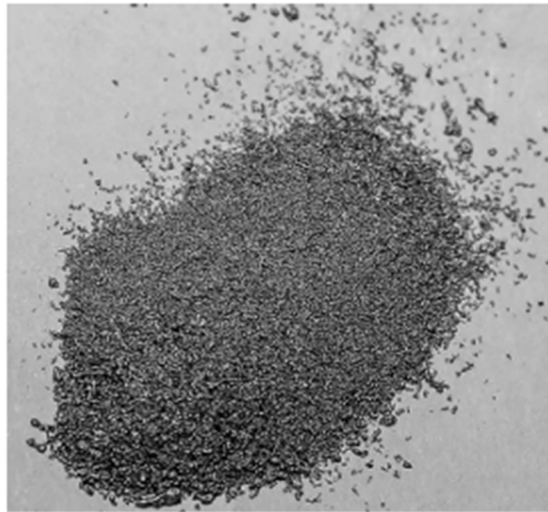


Figure 3. Al Powder



Figure 4. C Powde

Tools



Figure 5. Balance



Figure 6. Torque Wrench



Figure 7. Dies



Figure 8. Arch Welding Inverter

2.2 Procedure

Preparation of Starting Materials

The general flow of the project began with the preparation of starting materials, followed by balancing, mixing, and compaction. For the pure material sample, the ratio of TiO₂, Al and C was 3:4:3 without any excess. Other samples involved varying processing parameters by introducing TiO₂ and C in excess, specifically in quantities of 20%, 40%, 60%, 80%, and 100% excess. The mixing and compaction were performed using tools such as an Ohaus Advanturer Balance, a Torque Wrench, and Dies and Punch.

2.3 Combustion Synthesis

The combustion synthesis process was carried out in a combustion chamber with an atmospheric condition, initiated using an arc flame as the ignition technique. Arc welding was specifically used for the combustion process. The reactant powders, after being pressed into a pellet, are known as a green compact or simply green.

2.4 Product Characterization

The synthesized products were subjected to microstructure characterization. Testing of the materials was performed using Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) machines. This allowed for the observation of the microstructure and mechanical properties of the synthesized products. SAM was used to observe the particle shape and size, while XRD was used to detect the phase composition, such as TiC and Al₂O₃.

3. Results And Discussion

3.1 Synthesized Product

The synthesis of ceramic materials was conducted using various reactant compositions, starting with a pure material sample and then introducing TiO₂, Al and C in increasing excess amounts.

The pure material sample, composed of TiO₂, Al and C in a 3:4:3 ratio without any excess, exhibited the most rapid flame propagation upon ignition using the arc flame. A clear flashing was observed when the material began to react.

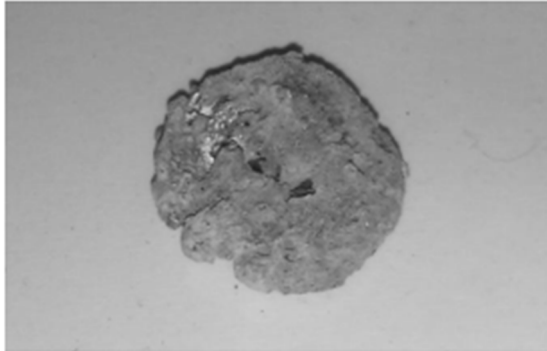


Figure 9. Synthesized product without Excess Addition

As the percentage of TiO_2 excess and C increased, the flame propagation rate decreased:

- 20% Excess: The flame propagation was still quick but not as rapid as the pure material.

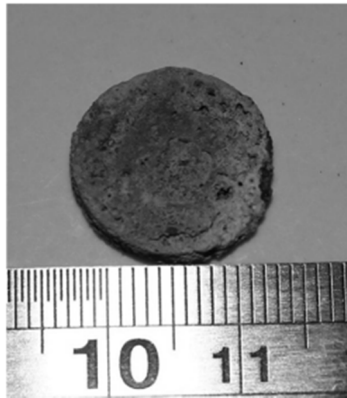


Figure 10. Synthesized product with 20% Excess Addition

- 40% Excess (Mass 1.288 g): The flame propagation was starting slow during the combustion process, and flashing appeared, indicating the material's reaction.

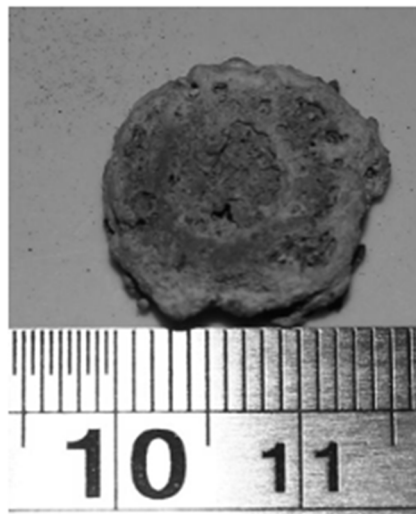


Figure 11. Synthesized product with 40% Excess

- 60% Excess: This sample showed slower flame propagation than the previous experiments, and flashing occurred when the material started to react. Notably, the resulting solid material could not be formed well, with half of the material retaining a powder appearance.

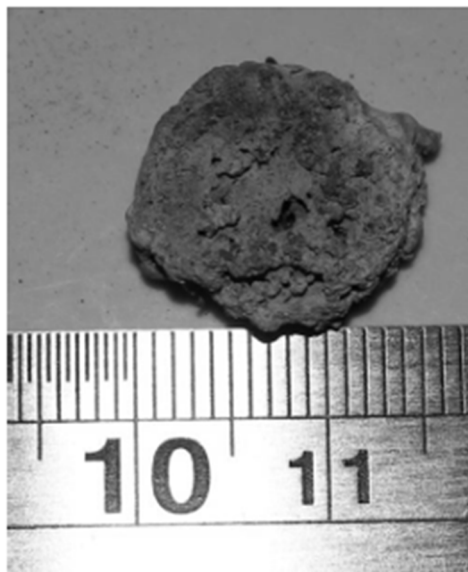


Figure 12. Synthesized product with 60% Excess

- 80% Excess (Mass 1.575 g): The combustion process was not successfully completed, as only a small part of the sample combusted.

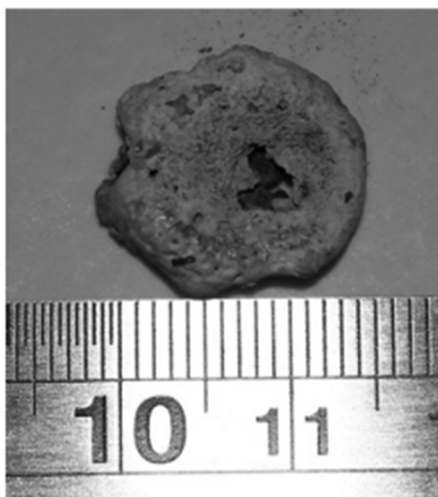


Figure 13. Synthesized product with 80% Excess

- 100% Excess (Mass 1.719 g): The material could not react finely during the combustion process, and no flame propagation was observed.

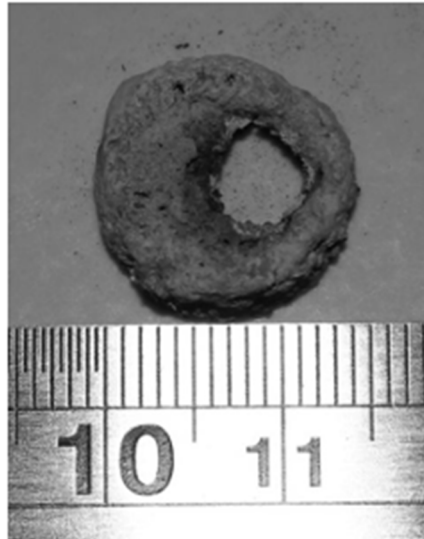


Figure 14. Synthesized product with 100% Excess

The results indicate that the reactant composition significantly influenced the ignition and flame propagation in the combustion synthesis process. The presence of excess TiO₂ and C appears to decrease the reactivity of the mixture, leading to incomplete combustion at higher excess percentages.

3.2 Phase Composition Analysis (XRD Pattern)

X-ray Diffraction (XRD) analysis was conducted to identify the phase composition of the synthesized products.

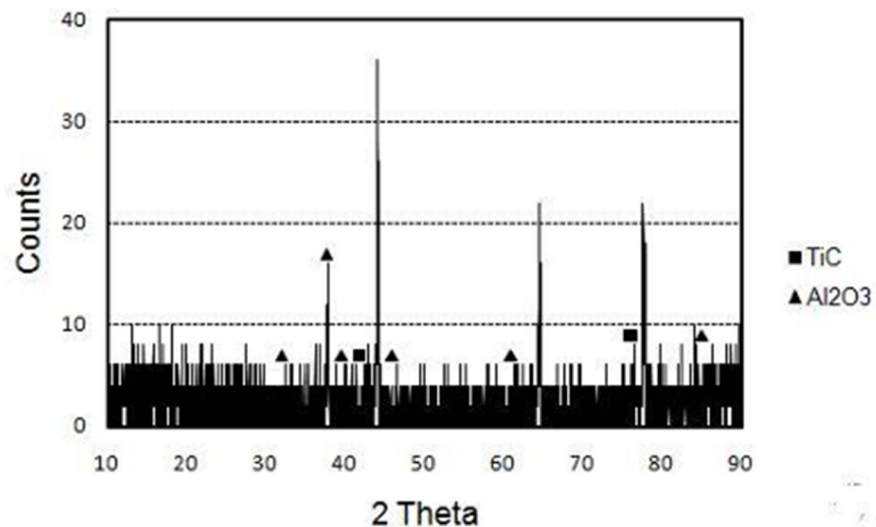


Figure 15. XRD pattern Without Excess

3.3 Pure Material (Without Excess)

The pattern of the pure material sample detected two points corresponding to TiC at approximately 45° and 80° in 2 theta. Al₂O₃ was detected at six points: 31°, 37°, 39°, 45°, 60° and 85° in 2 theta.

20% Excess of and

With the 20% excess addition, the number of detected TiC points increased from two to three, located at 35°, 41°, 72° and in 2theta . Al₂O₃ was detected at seven points : 19°, 31°, 37°, 45°, 60°, 37°, and 87° in 2 theta.

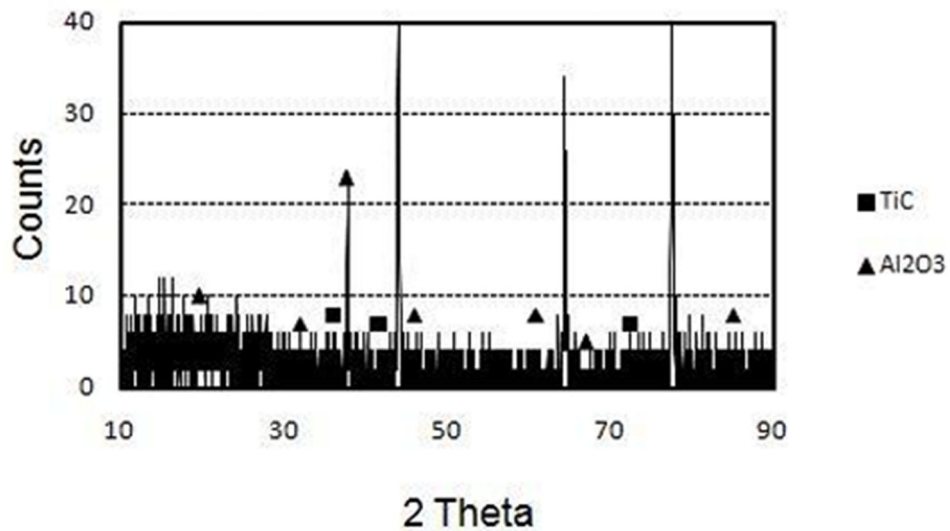


Figure 16. XRD Pattern With 20% Excess

40% Excess of and

The 40% excess sample showed 2theta values almost the same as the 20% excess sample, but with additional TiC detected at 60° and 78° in 2 theta.

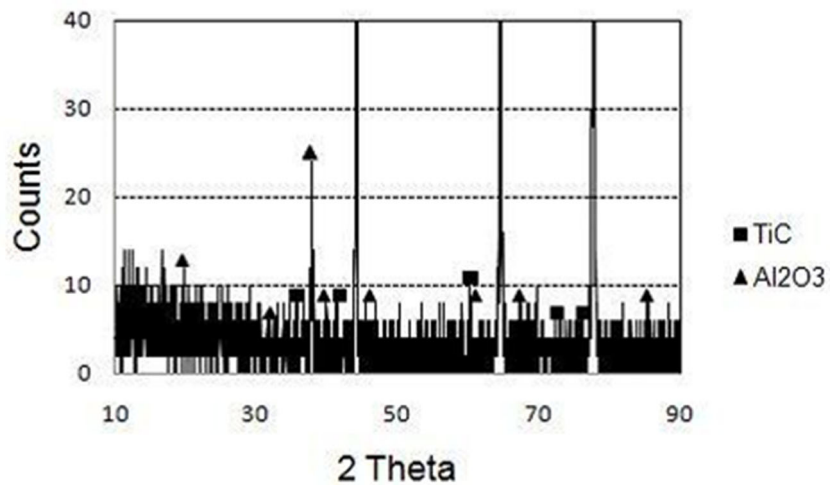


Figure 17. XRD Pattern With 40% Excess

The XRD patterns demonstrated that the increasing content of TiO₂ and C resulted in a higher peak result on the XRD test, which can be identified as the effect of the excess addition. However, it was noted that while Al₂O₃ was clearly seen, the XRD spectra for TiC did not appear on one of the products of the combustion process. This non-appearance of TiC in the reacted product was later attributed to the small size of the TiC particles.

3.4 Microstructure Observation

The microstructure of the synthesized product was observed using Scanning Electron Microscopy (SEM). The resulting particles were heterogeneous in shape and size. Some particles exhibited a dendritic shape, while others formed a cubic shape.

The effect of reactant composition on the microstructure was observed from the porosity of the reacted product. The results indicated that the increasing content of TiO₂ and C led to an increase in porosity. The differences in microstructures were visualized in the SEM figures for samples without excess, with 20% excess, and with 40% excess.

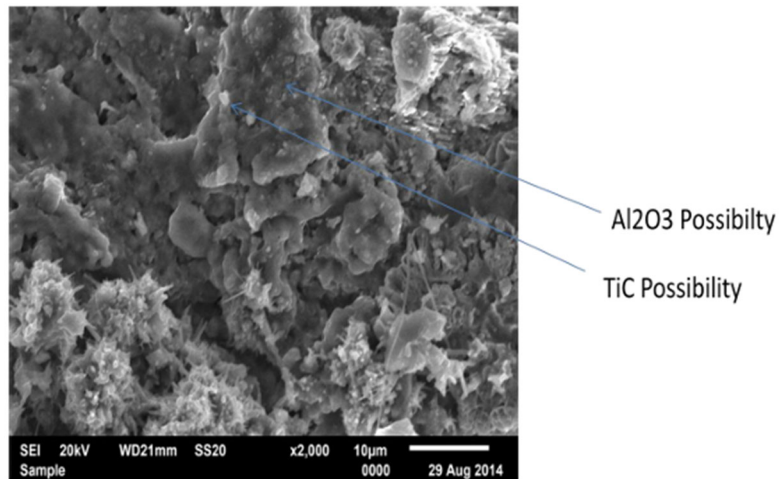


Figure 18. SEM Figure of Without Excess Synthesized Product

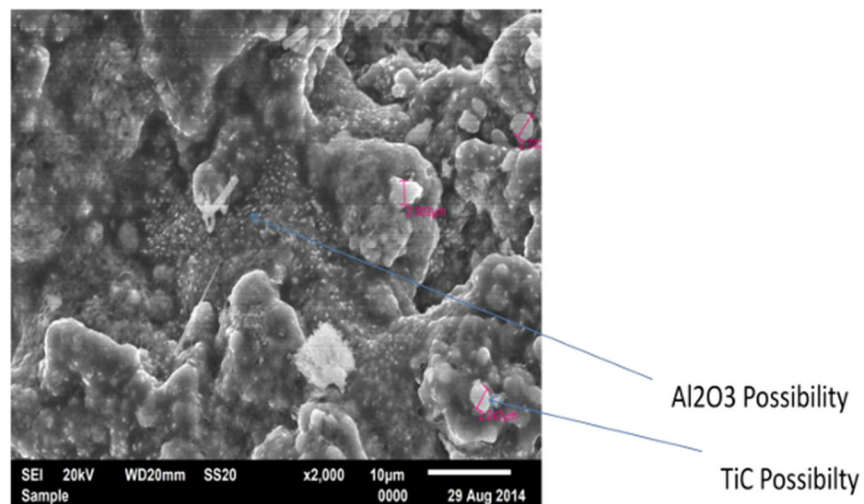
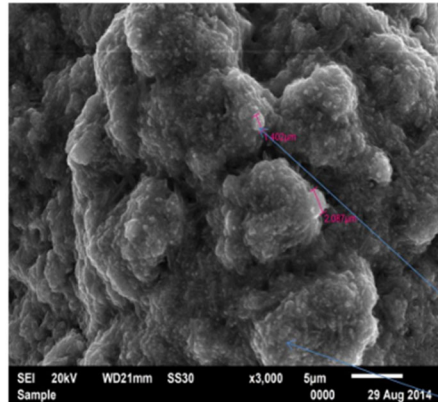


Figure 11. SEM Figure of With 20 % Excess

Figure 23.
With 20 %



SEM Figure of
Excess

TiC Possibility

Al2O3Possibility

4. CONCLUSION

4.1 Conclusions

Based on the analysis and discussion of the experimental results, the following conclusions can be drawn:

- The production of ceramic material using combustion synthesis was successful. The reactant composition significantly influenced the ignition and flame propagation.
- The phase composition of the synthesized product was detected using SEM and XRD. Al₂O₃ was clearly observed in the XRD pattern.
- The effect of reactant composition on the microstructure was observed through the porosity of the reacted product. The increasing content of TiO₂ and C resulted in an increase in porosity.
- The increasing content of TiO₂ and C can also be identified from the XRD pattern, as the more excess addition resulted in a higher peak on the XRD test.
- The XRD spectra of titanium carbide (TiC), one of the products, did not appear clearly in the reacted product. This is likely due to the small size of the TiC particles.

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