

Effect of Reinforcement Content and Milling Time on TiN-IN625 Composites

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Inconel 625 superalloy metal matrix composite reinforced with titanium nitride through powder metallurgy technique is an alternative for improving the mechanical properties of the well-known corrosion-resistant nickel alloy. In this paper, high-energy ball milling (HEBM) produced the TiN-Inconel 625 composite powders in a SPEX-type vibrating mill. The objective was to address the effect of the reinforcement content and milling time on the characteristics of the composite powders for posterior application on laser cladding. Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), Laser Diffraction (LD), and X-ray diffraction (XRD) characterized the samples. The results showed that HEBM provided an effective method for producing TiN-Inconel 625 composites due to the good integration of TiN reinforcement into the nickel alloy matrix.

Keywords: *Inconel 625, TiN, Metal Matrix Composites, High-Energy Ball Milling.*

1. Introduction

The material's surfaces are the most critical components in engineering, as they are subject to various phenomena such as fatigue, corrosion, and wear. A more noble material can be applied to the surface of substrates to enhance the properties and extend the service life of equipment and structures. Several techniques, including laser cladding, can be used to deposit protective coatings^{1,2}.

Nickel-based superalloys are widely used in applications where a combination of superior mechanical properties and excellent workability are required. Inconel 625 is a Ni-Cr-based austenite superalloy that presents a high chromium content, which improves the balance of creep performance, fatigue strength, and tensile while maintaining oxidation and corrosion resistance. Despite the good mechanical properties of the Inconel 625, due to the fast growth of modern industries, there has been an increasing demand for materials with even superior properties³⁻⁵.

Usually, metal matrix composites (MMC) improve the material's mechanical properties. Composites combine two or more materials with different properties, producing final characteristics different from the original constituent⁶. Several studies have been carried out on Inconel 625 matrix composites aiming to improve the service performance of components, especially regarding their hardness, wear resistance, and ceramic reinforcements. Most preceding works focus on reinforcement by using titanium carbide (TiC), tungsten carbide (WC), chromium carbide (CrC),

and carbon nanotubes⁷⁻¹⁰. Titanium nitride (TiN) is rarely explored in previous literature¹¹, although it is a promising candidate as reinforcement in the Ni coating due to its high modulus, good combination ability with Ni-base superalloy, and excellent thermal stability at high temperature¹².

Many fabrication techniques have been proposed to perform the synthesis of nickel matrix composites. The reinforcement with harder and temperature-resistant ceramic particles is considered attractive for use in MMCs due to its isotropic properties, easy fabrication, and low cost¹³. In this way, modern Powder Metallurgy (PM) is considered as a suitable technique to process composite materials. The microstructure, morphology and improvement in the mechanical properties of the resulting composites are affected by the intrinsic properties, size, volume fraction and distribution state of reinforcement. High-energy ball milling (HEBM), which is a specific PM technique of mechanical alloying^{14,15}, may be used to produce uniform dispersions of particles in nickel-base superalloys, since the deposition of fine ceramic-reinforced MMC clads is challenging because of the divergence of fine particles. In the HEBM process, a powder mixture is placed in the ball mill and subjected to high-energy collisions from the balls. The mixed powders are cold welded and fractured under high energy impact. The purpose behind is to attain a homogeneous mixing rather than breaking the matrix and reinforcements into fine grains^{16,17}.

This study explores the manufacture of the TiN-Inconel 625 powders by HEBM and the effect of reinforcement content and milling time. The main objective is to evaluate the incorporation of the TiN nanoparticle into the Inconel 625 matrix to allow the posterior deposition of the MMC clads.

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2. Experimental Procedure

Table 1 presents the Ni-based superalloy IN625 powder chemical composition used as the metallic matrix material. TiN ceramic powder was applied as material reinforcement. The composites’ compositions with the corresponding reinforcement content and milling time used in each sample for the HEBM are presented in Table 2. On top of the final mass, 2wt.% of C18H36O2 (stearic acid) was added to the milling process to act as a lubricant and anti-caking agent, working as a process controller agent (PCA).

A SPEX-type vibratory mill set at 720 rpm manufactured the TiN/Inconel 625 composites by a high-energy ball milling, one of the most frequently used systems for mechanical alloying due to the small amount of powder used. SPEX vibrating ball mill is characterized by a figure-8 motion. A container made of AISI 304L stainless steel was used, along with 100C6 steel spheres (1% carbon, 1.5% chromium) with an average diameter of 6.4mm and isopropyl alcohol as PCA. The ball-to powders weight ratio (BPR) was 10:1 and milling times of 60 and 120 min evaluated the incorporation of the reinforcement in the metal matrix.

After milling, the powders were dried at 100°C to remove alcohol residues.

For the characterization of the powders produced, X-ray diffraction (XRD) investigated the phases in the TiN-IN625 composites, covering a range of 20° to 100°, with a scanning rate of 0.02°/s, under a voltage of 40 kV and a current of 30 mA, performed on Shimadzu Maxima XRD – 7000 equipment. The morphology was analyzed using a TESCAN MIRA 3 - Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS). The particle size was measured by Laser Diffraction (LD)^{18,19} using Malvern Mastersizer 2000. The samples were suspended alcohol and subjected to ultrasonic agitation, covering a size range from 0.02 µm to 2000 µm.

3. Results and Discussion

3.1. Granulometry

Figure 1 presents the particle size distribution of the starting powders and the composite powders generated by HEBM.

Firstly, the unreinforced IN625 powder curve can be noticed as a monomodal shape with a symmetrical curve, presenting an average particle size distribution of approximately 150 µm. On the other hand, for the reinforcement powder of TiN, the curve was multimodal, and the average particle size was 20 nm. Due to the high difference in the fine particles of the starting powders, composite powders by HEBM were fabricated.

The HEBM process promotes a change in particle size distribution, according to the curves shown in Figure 1 (c-h). The curves for low milling times exhibit a slight bimodal shape. For 120 min, the curves presented a monomodal shape²⁰. However, the reinforced powders with higher milling time presented an asymmetric distribution, with a slight tendency towards the largest particle size region. This phenomenon indicates that cold welding occurs more strongly than fractures of the powder particles²¹.

Figure 2 shows the median particle sizes of the produced powders. The TiN powders present a small scale and have no difference in average particle size.

There is a considerable increase in particle size for the reinforced powders at a low milling time. On the other hand, there is a decrease in average particle size when the milling time increases. The effects of the cold welding of particles were overcome by the fractures, reducing the particle size, and the equilibrium state between welding and fracture was achieved^{22,23}. However, the objective was to incorporate the reinforcement into the metal matrix, making it unnecessary.

Regarding reinforcement, it is important to note that when the amount of TiN in the Inconel matrix is low, an increase in TiN content leads to a reduction in the size of the powder particles. This phenomenon is a consequence of the fracture resistance of the matrix. However, larger particles were found when the reinforcement concentration in the matrix increased from 5% to 10%. The reinforcement promoted an increase in cold welding during the early stages of HEBM²⁰.

3.2. Microstructural characterization

Figure 3 (a-b) and Figure 4 (a-f) show the morphologies of the starting powders and the milled powders with different reinforcement and milling times, as described in Table 2, respectively.

Table 1. Chemical composition of Inconel 625 powder.

Weight percent wt% (nominal)					
Ni	Cr	Mo	Nb	Fe	Other
58-63	20-23	8-10	3-5	<=5	<2

Table 2. Composition and nomenclature of composite samples.

Samples	Composition	Milling time
1	IN625 + 2% TiN	60 min
2	IN625 + 2% TiN	120 min
3	IN625 + 3% TiN	60 min
4	IN625 + 3% TiN	120 min
5	IN625 + 5% TiN	120 min
6	IN625 + 10% TiN	120 min

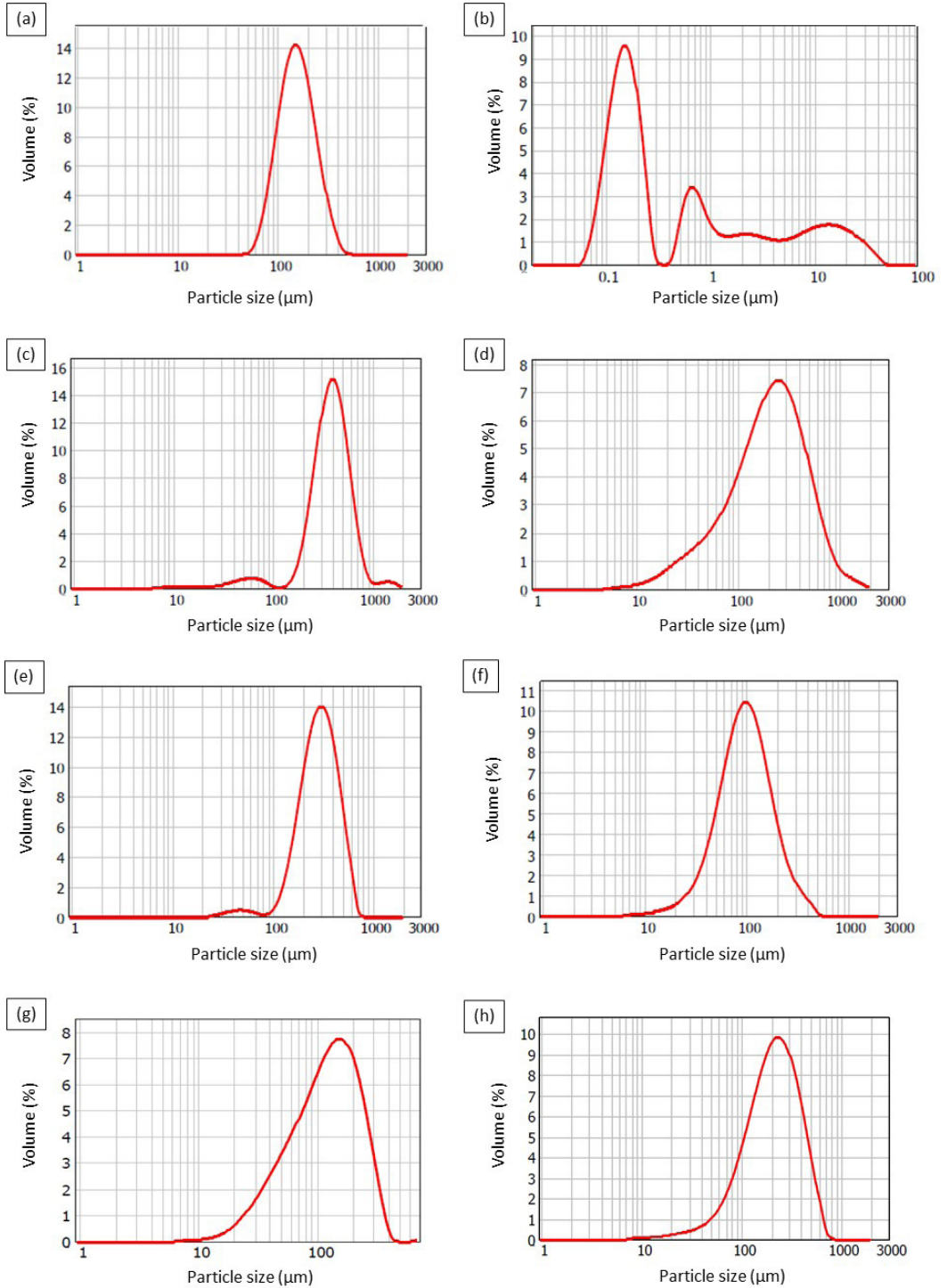


Figure 1. Particle size distribution of the starting powders of (a) IN625 and (b) TiN, and of the composite powders: (c) sample 1, (d) sample 2, (e) sample 3, (f) sample 4, (g) sample 5 and (h) sample 6.

The argon gas atomized IN625 powders are spherical of ductile materials, while the powder morphology of TiN is predominantly irregular and associated with the morphology of hard materials. On the other hand, for the composite powders, the spherical morphology of the original IN625 powder was altered to a more lamellar shape by the ball milling process

due to the continuous collisions between the particles and the balls. The powders were milled into flat pieces, regardless of the reinforcement content and milling time.

Figures 5 and 6 present EDS mapping for the 2% TiN/IN625 composite in order to evaluate the effect of milling time on the incorporation of the reinforcement.

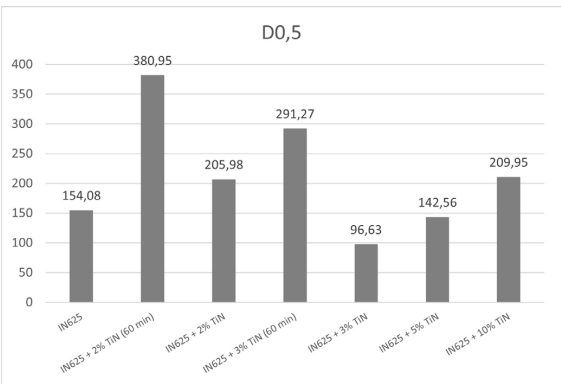


Figure 2. Median particle size of powders.

For a 60-minute milling time, there was not enough time for the fracture of the particles. The stage of cold welding prevailed, leading to the distribution of the fragile particles on the surface of the ductile material and an enhancement in the particle size of the powder composite, as presented in Figure 2. Increasing milling time promoted a reduction in particle size by the fracture stage and the presence of hard particles of the reinforcement powder was observed on the IN625 surface.

Figure 7 presents the elemental composition of the powders produced. It is the majority presence of Ni and Cr, which composes the matrix of IN625, as well as the incidence of other elements of the superalloy as Mo, Fe, and Nb. The presence of Ti in the final composite powders is observed.

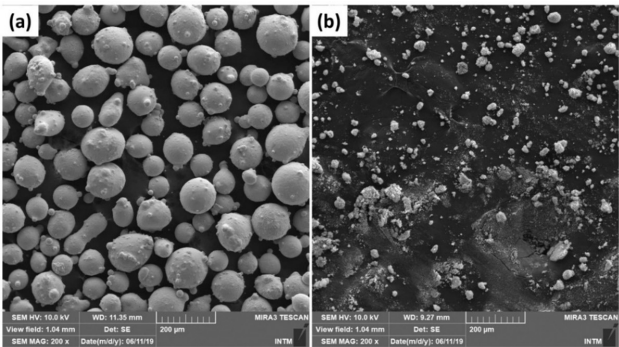


Figure 3. SEM images showing the morphology of the starting powders: (a) Inconel 625 and (b) TiN (200x).

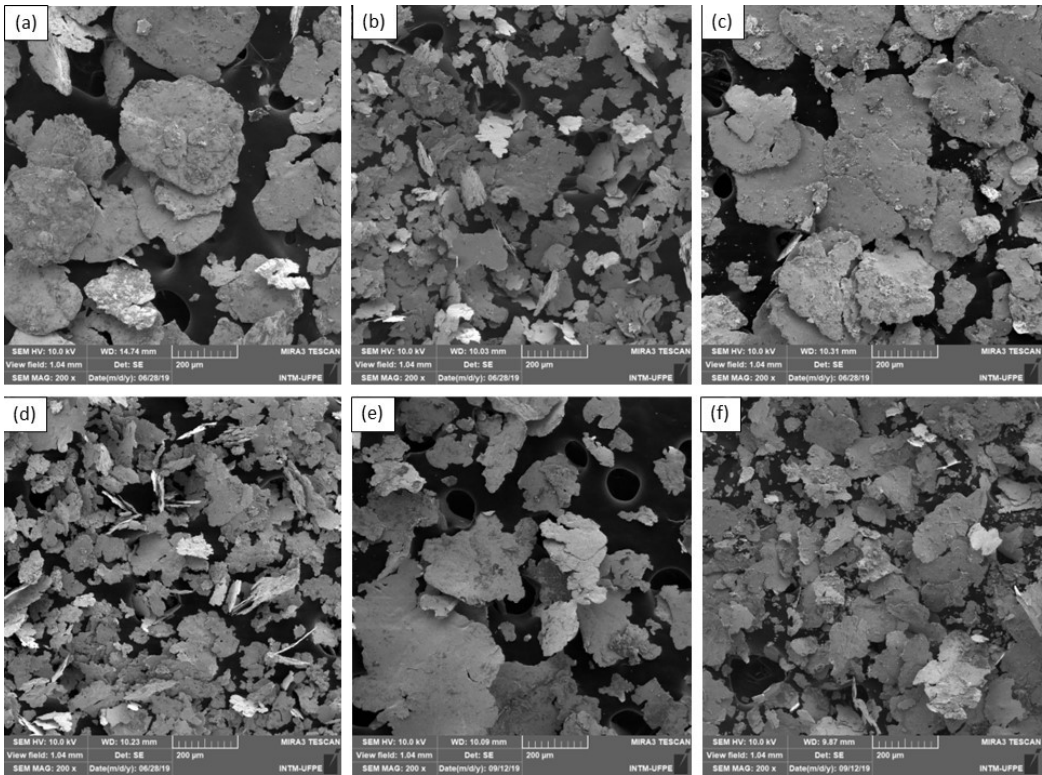


Figure 4. SEM images of the composite powders: (a) sample 1, (b) sample 2, (c) sample 3, (d) sample 4, (e) sample 5 and (f) sample 6.

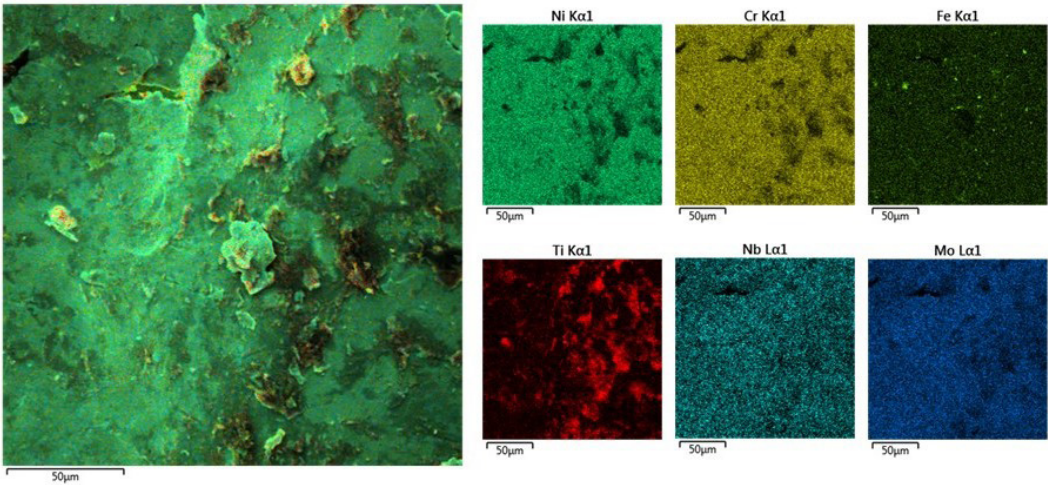


Figure 5. EDS mapping of the powder composite of 2%TiN/IN625 for a milling time of 60 min.

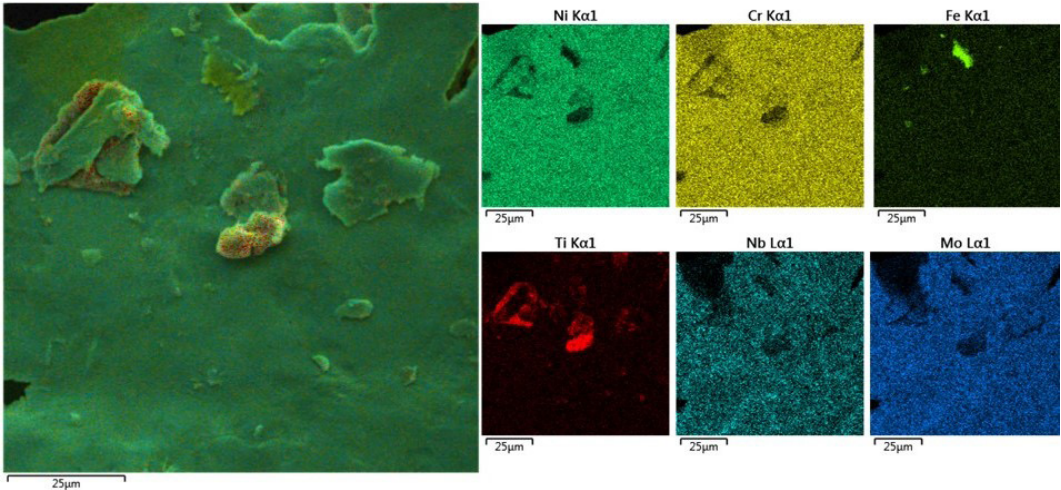


Figure 6. EDS mapping of the powder composite of 2%TiN/IN625 for a milling time of 120 min.

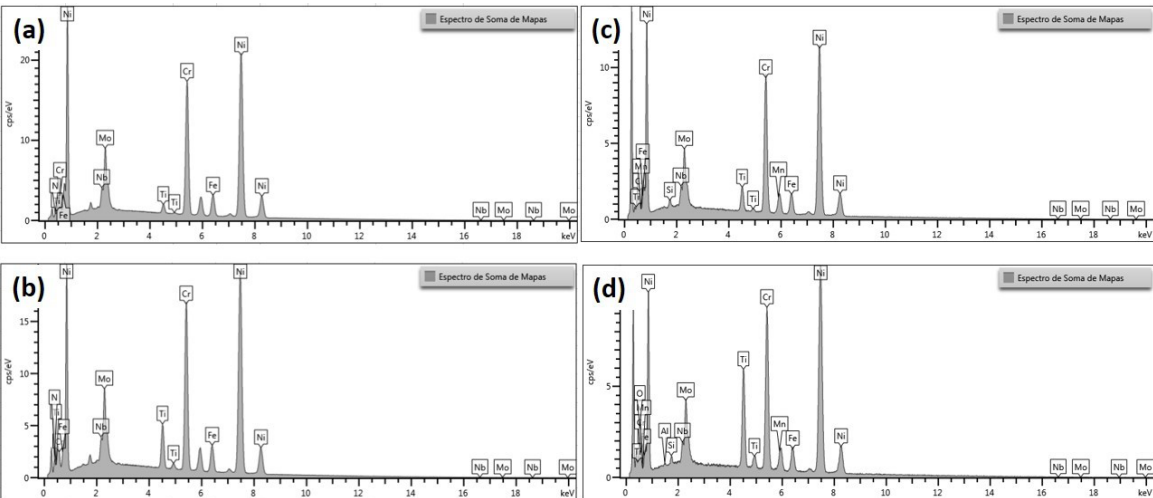


Figure 7. Elemental composition of the TiN-IN625 composites with a reinforcement content of (a) 2%, (b) 3%, (c) 5% and (d) 10% for 120 min milling time.

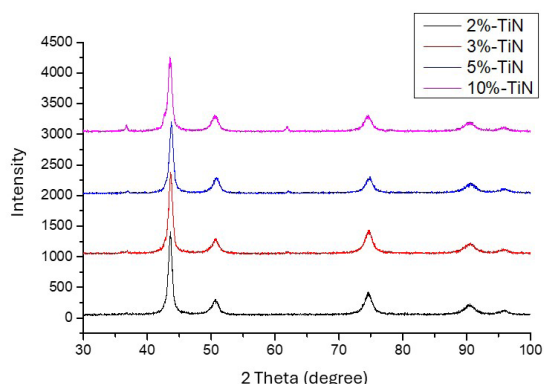


Figure 8. XRD patterns of the TiN/IN625 composites.

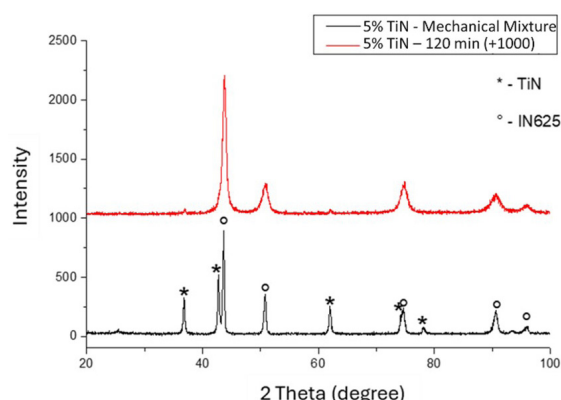


Figure 9. XRD diffraction pattern of the 5wt%Ti-IN625 powder milled and mechanically mixed.

3.3. Phases identification

Figure 8 presents XRD patterns of the starting powders of Inconel 625 and TiN and the TiN/IN625 composites with different reinforcements for 120 min of milling time.

The XRD spectrum of composite particles were found at 2 theta angles of 43.6°, 50.8°, 74.6°, 90.5° and 95.8°, which belongs to the nickel matrix γ and can be indexed to the diffraction of (111), (200), (220), (311) e (222) planes. The standard of the pure Ni from the ICDD- database are 44.5°, 51.9°, 76.4°, 92.9° and 98.5°, which are slightly higher than the ones found for the IN625 alloy. This difference in lattice parameter can be associated with the effect of alloying elements of IN625 and the dislocations in the crystal. Then, the crystal structure of γ phase in IN625 superalloy and TiN-IN625 composites is easily distorted owing to the large number of alloying elements in material²⁴. No phases other than the FCC γ - Ni phase with lattice parameters close to pure Ni could be detected. Therefore, the phases of the TiN-IN625 composites did not change in comparison to the IN625, except for the introduction of TiN particles.

In the IN625 + 10% TiN composite, small peaks can be observed at 36.7°, 42.6°, 61.8°, 74.1°, and 78°. These peaks correspond to the diffraction patterns of the (111), (200), (220), (311), and (222) planes of TiN, respectively.

Although peaks related to TiN are relatively small, their intensity increases with higher reinforcement contents. The observed reduction in peak intensity suggests that the TiN particles were incorporated into the IN625 matrix.

To confirm the incorporation of the TiN particles, due to the difference in particle size distribution and limit detections of the equipment, XRD patterns were performed on the mechanical mixture and compared with the milled powders to observe if the nanoparticle of TiN can be detected by the equipment. Figure 9 presents the comparison for the IN625 + 5% TiN.

The intensity of the peaks of TiN is easily highlighted for the mechanical mixture, meaning that it can be effectively detected by the XRD. The intensity of the peaks reduces considerably with the milling process, which is higher as the content of reinforcement increases. Therefore, the incorporation of the TiN in the milled powders is confirmed.

4. Conclusions

The following conclusions were observed:

- 1 - High-energy ball milling (HEBM) is an effective means of producing TiN-Inconel 625 composites and allows the TiN reinforcement to be well incorporated into the nickel alloy matrix;
- 2 - The milling process was successful as the particle size distribution curve presented a monomodal shape;
- 3 - An increased reinforcement insertion into the matrix decreases the particle size due to the lower fracture resistance, although cold welding rates in the early stages of HEBM may occur for higher reinforcement contents.

5. References

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Data Availability

The entire data set supporting the results of this study was published in the article itself.