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H. Murat Enginsoy, Emin Bayraktar, Dhurata Katundi, Ibrahim Miskioglu. Detailed investigation of TiN effect on hybrid intermetallic composites manufactured by combined sinter + forging method. Mechanics of Advanced Materials and Structures, In press, pp.1-12. 10.1080/15376494.2021.1886380 . hal-03140011

HAL Id: hal-03140011

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Mechanics of Advanced Materials and Structures

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/umcm20

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To cite this article: H. Murat Enginsoy , Emin Bayraktar , Dhurata Katundi & Ibrahim Miskioglu (2021): Detailed investigation of TiN effect on hybrid intermetallic composites manufactured by combined sinter+forging method, Mechanics of Advanced Materials and Structures

To link to this article: https://doi.org/10.1080/15376494.2021.1886380

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MECHANICS OF ADVANCED MATERIALS AND STRUCTURES
https://doi.org/10.1080/15376494.2021.1886380

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ORIGINAL ARTICLE

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Detailed investigation of TiN effect on hybrid intermetallic composites manufactured by combined sinter + forging method

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ABSTRACT

Hybrid intermetallic composites (HICs-aluminum based matrix, new hybrid design) reinforced with titanium nitride (TiN) and alumina (γ-Al_2O_3) were designed and manufactured through the innovative combined method of sinter + forging. This method results in highly dense materials with, in general, improved mechanical properties. In the composites TiN content was varied (10, 20 and 30 wt %) but γ-Al_2O_3 content was kept constant at 10 wt%. Additionally, 5 wt% each of Cu and Ni were mixed in to improve processing of the composites. The mechanical properties were specified by different destructive test methods (wear performance, micro hardness, quasi-static compression, three-point bending tests). Wear performance was investigated by scratch experiments. Scanning electron microscopy (SEM) was used to determine the microstructure, interface characteristics and the fracture surfaces. The SEM analysis yielded that TiN powders were located at the grain boundaries and this can be attributed to the improvement observed in the mechanical properties and wear performance with the addition of TiN.

ARTICLE HISTORY
Received 28 September 2020
Accepted 2 February 2021

KEYWORDS
Electron microscopy; forging; mechanical properties; mechanical testing; metal-matrix composites (MMCs); fracture; microstructural analysis; microstructures; sintering; wear

1. Introduction

The motivation behind designing composite materials is to produce highly efficient (high specific weight/stiffness, good fatigue and tribological characteristics), multi-functional materials that industry is demanding. In a typical design of a composite the synergistic effects of the composite's constituents are exploited to come up with a new material with properties tailored to the requirements of the application in hand. Recent research has shown that metal matrix composites (MMCs) reinforced with ceramic particles [1–4] can satisfy the requirements for lightweight, high specific weight/stiffness and good wear characteristics sought for in different industries ranging from aviation, automotive to manufacturers of recreational goods [3–14]. Among the matrix materials for MMCs, aluminum is a preferred one because of its low density, good thermo-mechanical properties and also ease in manufacturability [3–9]. Aluminum matrix composites (AMCs) exhibit good physical and mechanical properties and they can easily be produced by conventional manufacturing processes. These attributes make AMCs very attractive new materials that can replace structural components, and they have been successfully used to that end in aviation and automotive industries. Of course, as more AMCs with new constituents along with low cost production methods are developed, the use of these in industry is expected to increase further [2].

Today, new generation of aluminum matrix composites with a variety of particle reinforcements are being produced by different manufacturing methods and the effect of reinforcements on the physical and mechanical properties of the AMCs are evaluated in detail with the goal of developing AMCs with properties suitable for structural applications [15–17]. In this research, AMCs reinforced with titanium nitride (TiN) and alumina (γ-Al_2O_3) are investigated with the aim to be used as linkages, connectors in aeronautical applications where high hardness and high wear resistance along with high resistance to compression and bending loads are needed.

The interactions of the reinforcing elements with the aluminum matrix depend on their chemical structure, but they are also strongly dependent on the manufacturing method. Factors such as temperature and pressure used during manufacturing method can have a great effect. Many similar studies have been done in the literature but different chemical treatments and/or reactions were used. For example, TiC ceramic particles have been reacted in situ in the aluminum alloy composites. Also, TiN particles were generally cold sprayed in aluminum composites (e.g. AA 2319). In many of the studies, fine ceramic reinforced aluminum composites were generally produced by reactive metal infiltration and/or by melting process very similar to in situ reaction [17–23]. Based on our scientific and experimental knowledge

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and our agreement with the aeronautic research team, we have been developing the method "Sinter + Forging and/or Thixoforming," for the manufacture of hard ceramic particle reinforced composites. We combine two processes directly, i.e. simple powder metallurgy route is followed by the final processing as hot forging and or thixoforming as we have explained in detail in our former publications e.g. [2, 4, 5, 7–9, 13–18]. Again, we have been developing this method for mass production of the connection (knuckle) parts of different sizes in aeronautical/aerospace engineering applications as a low cost and efficient manufacturing process. Naturally, the lower cost of the manufacturing method has a special importance for the industry. For these reasons, the determination of optimum parameters is always taken as the main objective in the development of such materials.

2. Materials and methods

2.1. Manufacturing process

The manufacturing of composite material specimens presented in this research was carried out by using powder metallurgy via combined the innovative process of Sinter + Forging. In the first stage of manufacturing, each component of the hybrid metal matrix composite was mixed together. The stage in this process was carried out by milled and homogeneous distribution in a pulverizing device for 2 hours under the oxidation protection. After these processes, ball milled was carried out for 1 hour under 4000 rpm. The powder mixture obtained as a result of these processes was compressed under a pressure of 250 MPa. The compacts obtained from the compressed powders were sintered (under an argon inert gas atmosphere at 600 °C for 45 min), followed by forging (at 500 °C). After the combined method mentioned above, this process we have explained here is called: "Sinter + Forging." This innovative manufacturing method has significant advantages compared to traditional methods. For example, it has important advantages such as low cost-fast manufacturing style, ability to manufacture complex designs in complex ways and ease of use of equipment/systems used in the manufacturing process.

Constituents of hybrid AMCs specimens manufactured in three main categories were presented in Table 1 and Figure 1 presents the general characteristics of fine powders received from Sigma Aldrich France. The particle size density of these powders was Al ($\rho = 2.7\,g/cm^3$, US mesh = 625 with $d = 20\,\mu m$), γ-Al_2O_3 ($\rho = 2.83\,g/cm^3$ with $d = 1–3\,\mu m$), TiN ($\rho = 5.22\,g/cm^3$, $d = 1–3\,\mu m$), Cu ($\rho = 7.96\,g/cm^3$, $d = 1–2\,\mu m$) and also Ni ($\rho = 7.9\,g/cm^3$, $d = 1–2\,\mu m$) respectively. The particle size information was provided by the French industrial partner, and it was specified that more than 80 to 90% of the particles in the batch were of their respective size given above.

Table 1. Constituents of hybrid composites prepared in three main categories (wt %).

Specimen category	Al matrix (wt %)	γ-Al_2O_3 (wt %)	TiN (wt %)	Cu (wt %)	Ni (wt %)
TiN-I	70	10	10	5	5
TiN-II	60	10	20	5	5
TiN-III	50	10	30	5	5

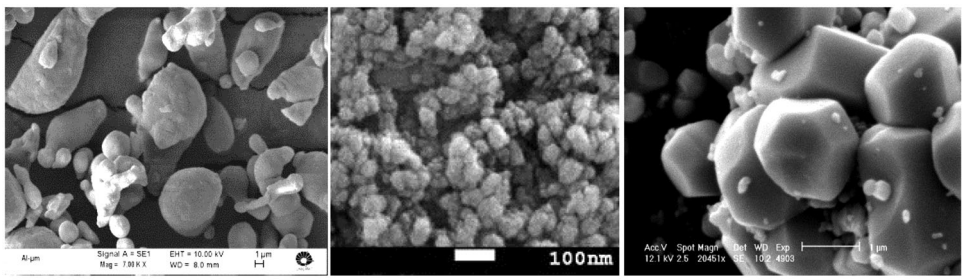
2.2. Experimental analyses

2.2.1. Microstructural analysis procedures

In the composite specimens manufactured, Scanning Electron Microscope (SEM) was used for the analysis of the micro scale internal structure. The observation and evaluation of reinforcement distributions within the matrix and matrix/reinforcement interfaces were performed through SEM. For all composite specimens, X-Ray Diffraction (XRD) measurements and density measurements (Archimedes method) were made.

2.2.2. Destructive material test procedures

Destructive test types were used to determine the mechanical behavior of composite materials developed within the scope of the study. Appropriate test specimens were prepared according to the standards of the respective test types. In this context, wear performance, micro hardness, quasi-static compression and 3-point bending tests were conducted. The wear performances of composite specimens were investigated through the zirconium ball indenter (under 1.9 N force, at 15 Hz frequency, in 50×10^3 and 100×10^3 cycle repetitions). The tangential and normal forces arising during the experiment were recorded with the LabView software we developed, and the friction coefficient was calculated from the ratio obtained from these parameters [24, 25]. In order to reveal the wear performance of composite samples more clearly, the determination of the wear damage, the amount of wear and the depth of wear on



Aluminum γ-Al2O3 TiN

Figure 1. Powders used in this work; Al, γ-Al_2O_3 and TiN received from French industrial partners.

the sample at the end of the scratch tests were done in three dimensions by using the optical roughness meter (Surf-Scan). The scratch test device enhanced in SUPMECA-Paris laboratory was used to determine the wear performance of the composite material manufactured. Micro hardness tests (HV0.5) were performed in order to conduct further research on the internal structure of the developed composite material on a micro scale. Specimens to be measured were first dimensioned with precise cut. Then polishing was applied to the surface to be examined. The specimens, whose surface polishing is finished, were taken into compacted and then etching in acid was applied in order to observe the formations (grain boundaries, grain sizes, etc.) in the inner structure more clearly. At the end of this process, micro scale tests were performed by making measurements from different regions such as interface, reinforcement and matrix on each specimen prepared.

Three-point bending (3PB) and quasi-static compression tests were conducted. Quasi-static compression tests were carried out according to the DIN 50106 standard. This test specimen sizes were prepared to be in cylindrical form at height/diameter ($H/D \geq 1.5$). Constant test speed has been chosen as 1 mm/min according to the relevant standard. Average values were obtained by repeating the experiments 3 times for each specimen composition. Three-point bending tests were carried out according to the ASTM D 790 standard. In this context, the specimen geometry has been prepared to be rectangular form.

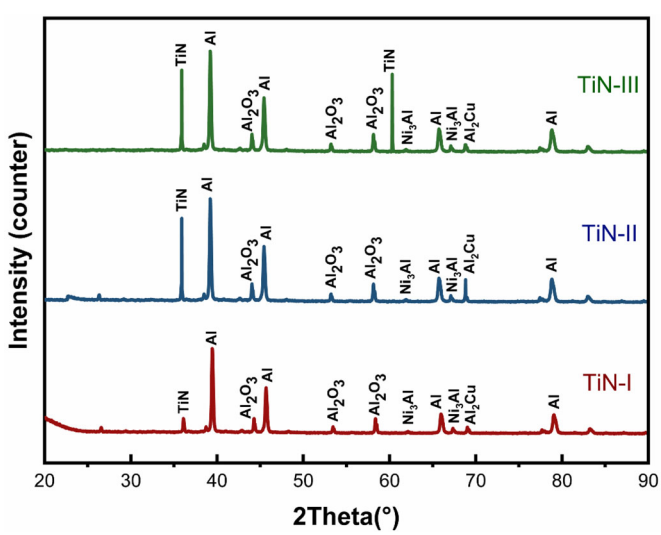


Figure 2. XRD diagrams for TiN-I, TiN-II, TiN-III specimens.

3. Results and discussions

3.1. Comprehensive analyses of microstructure

Mixing of the constituents by high energy milling very often yields a fusion phase. It was not clear in the XRD shown in Figure 2, but in the early stage (about 2Theta ~30°), TiAlN should be detected, but we think that this is due to low sintering temperature that was used in this study. The existence of the intermetallic phases confirmed that phase forming solid state reactions began throughout the mechanical fusion process. Here two typical intermetallic phases, Al_2Cu and Ni_3Al were observed in all compositions around 2Theta 60°–69° after a natural heat treatment around at a temperature of 450–475 °C during processing of the composites. For TiN-I and TiN-III the intensity of the peak is very small, for TiN-II it well defined. All of the XRD measurements have been shown for three compositions in Figure 2 to justify the phases occurred during the processing of the composites (Sinter + Forging).

In our experimental conditions, this mechanical fusion occurred between particles and matrix was given in Figure 3 as an example for the composite of TiN-I. In this picture only some of the reinforcements were joined with the aluminum matrix. It should be considered that EDS spectrum shown is from the entire picture.

The densities of the three composites were measured by using Archimedes method. The initial green density has ranged from 85% to 95%. After final manufacturing of the composites, the densities were varied from 2.83 and 2.85 up to 2.87 g/cm^3.

As for the microstructures of the composites in three groups, they are presented in Figure 4 as low and high magnifications. TiN is always found a second phase in the aluminum matrix, scattered homogenously at the grain boundaries. For this reason, it never interacted with the aluminum matrix during sinter forging at the process temperature that we have applied (500–600 °C). However, it stimulated chemical bonding diffusion of the other particles with matrix. In Figure 4, the microstructures given for three

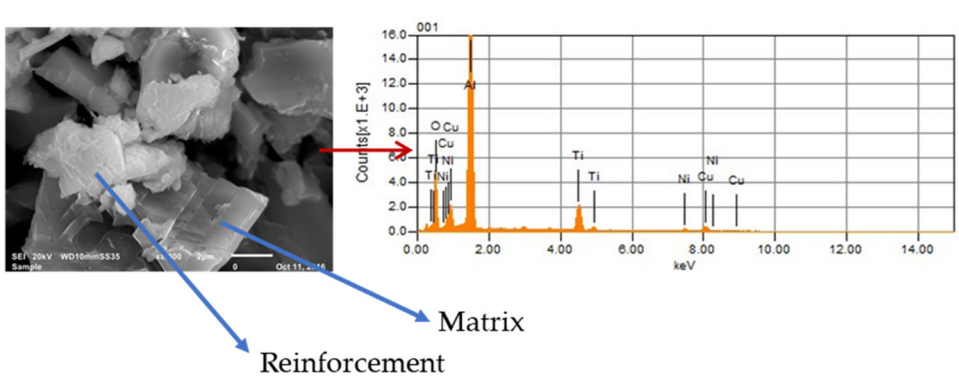


Figure 3. Mechanical fusion occurred between particles and matrix for the composite of TiN-I during high energy milling.

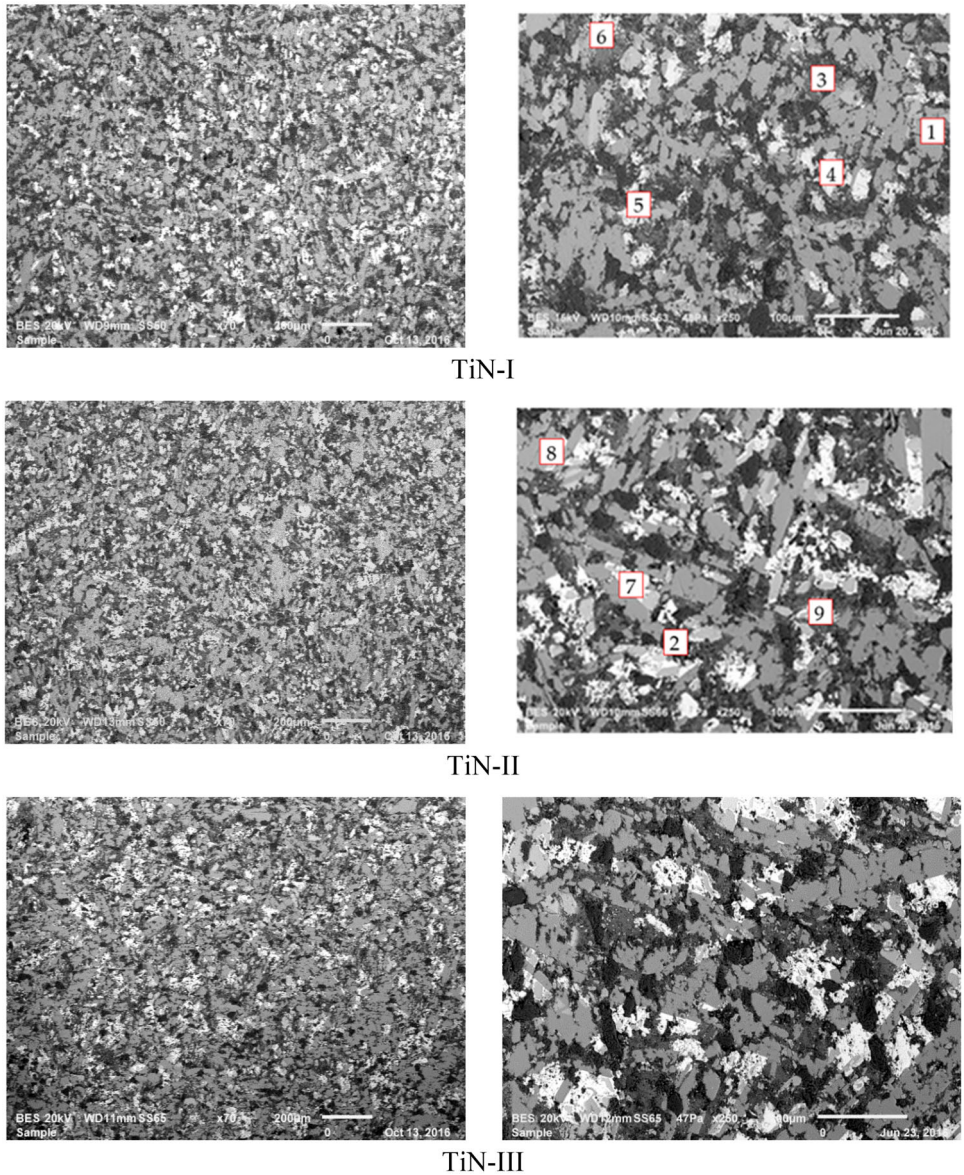


Figure 4. General microstructures of the three composites designed in this work, TiN-I, TiN-II and TiN-III, respectively in lower (left) and higher magnifications.

compositions justify this idea. Aluminum in the picture is seen as light gray (1) and γ-Al_2O_3 is seen as black (2) and dark gray (3) was estimated as TiAlN, white color (4) is seen as Al_2Cu and TiN mixed. However, TiN were distributed all in the microstructure as very fine particles as indicated in (5) and/or (6). Chemical bonding diffusion is seen in certain zones, one may indicate that in (7) and (8) or (9) in the pictures. The combined method, sinter + forging, applied in the present work improved this chemical bonding diffusion mechanism. As shown in the pictures, all of the reinforcements were well distributed in the matrix by means of the combined process (sinter + forging) applied for these composites.

In addition, it was observed that copper and nickel helped with the chemical bonding diffusion and forming of certain intermetallics, at the interfaces between the matrix and the reinforcements. As indicated in the former section, an intermetallic phase, Al_2Cu, in white colors sometimes in needle form occurred in regular way during the sinter + forging process of the composites. Figure 5 gives this very descriptive idea on the diffusion and forming of intermetallic phases at the interfaces between matrix and the reinforcements at the grain boundaries for the three composites. In the pictures, the gray part is the matrix, and the white part is Al_2Cu and TiN mixed with some traces of Ni and γ-Al_2O_3. The mutual chemical diffusion bonding tendency can be enhanced with the addition of copper and nickel to the aluminum matrix composites. Chemical diffusion bonding capacities of copper and nickel atoms increases with temperature and time due to the fine TiN particles with highly activated surfaces. For copper, this diffusion bonding can be easily seen in the range of 600–700 °C where the probability of copper forming intermetallic phases with aluminum is higher than nickel. Similarly, nickel could also form intermetallic phases with aluminum at higher temperature, around at 800 °C with longer processing time (NiAl,

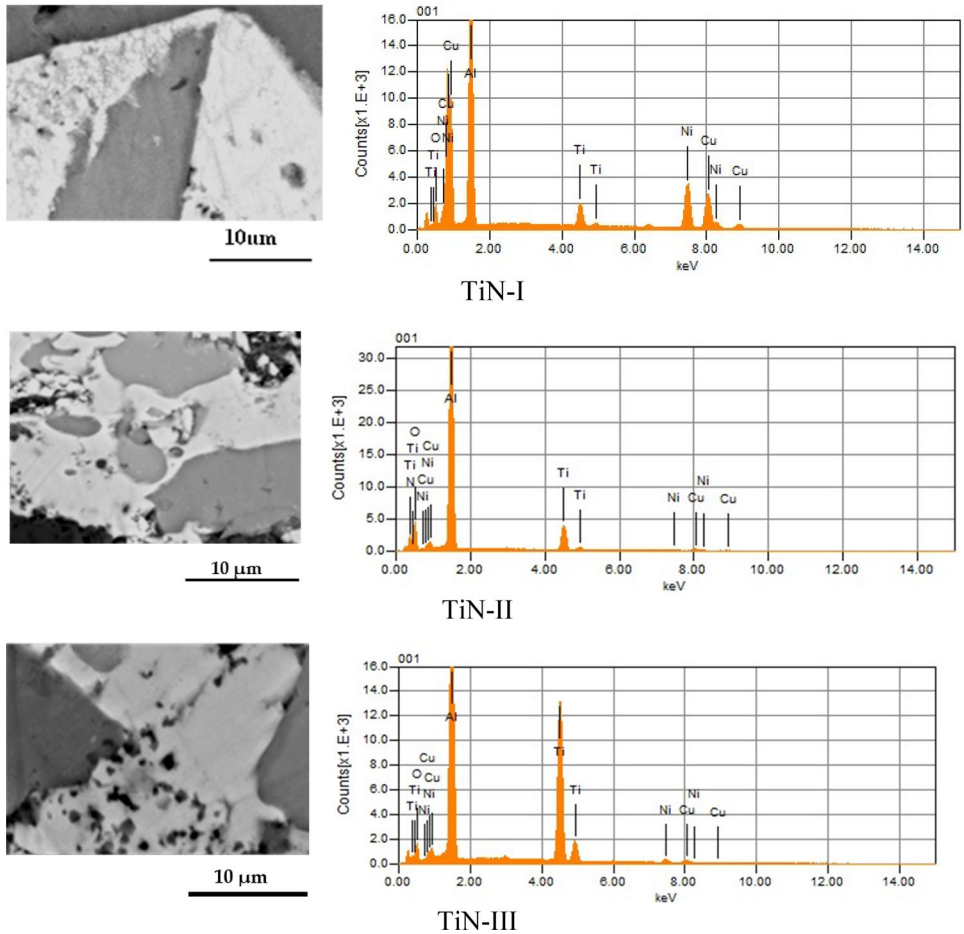


Figure 5. Diffusion and forming of intermetallic phases at the interfaces between matrix and the reinforcements at the grain boundaries for the three composites, TiN-I, TiN-II and TiN-III, respectively.

etc.). As it is well understood, the mutual chemical diffusion bonding tendency (atomic diffusion) from copper and nickel sides to the matrix, mainly to "TiN" side is greater than that in the opposite direction. Therefore, the copper and nickel could boost the chemical diffusion bonding tendency in this matrix and very often we observe a hybrid microstructure with graded composition in these composites. These typical diffusions at the interfaces were presented with EDS analyses in Figure 5 for three composites were performed on the whole picture, TiN-I, TiN-II and TiN-III respectively.

In general, the TiAlN phase structure is known for its dense morphology and fine grain structure that should be an obstacle for dislocations to move which improves the micro-hardness of the composite structure. The reason for the occurrence of the TiAlN phase structure in the present composite was most probably related to the critical amount of aluminum atoms embedded in the TiN lattice during the sintering process. We believe that this critical solubility of the aluminum should diffuse in metastable cubic TiAlN phase structure. Verification of this chemical reaction needs high resolution phase analyses with a Transmission Electron Microscopy (TEM). However, at this stage of the present research TEM analysis was not used which could be performed in future research.

A detailed analysis using SEM screening and EDX mapping (for copper, titanium and aluminum) to evaluate the distribution of the reinforcement elements in the matrix phase and their effects on phase formation is given in Figure 6. The mechanism of operation of the map analysis software is based on a color assessment showing that the mapped component (Al, Cu or Ti) is 100% available. The composition of the components decreases as the colors darken, while black indicates 0%. In the mapping analysis illustrated in Figure 6, the homogeneous distribution of very fine TiN particles (last line very fine particles in white color) are given and observed. In addition, the typical, usually needle-shaped homogeneous distribution of the Al_2Cu intermetallic phases in the matrix (midline) is also observed. The aluminum matrix contains all the reinforcement particles, so that a mixture of lighter colors dominates the mapping (first line). Still, white particles (100% aluminum) appear to be dense at the top of the picture. This means that a certain amount of copper (Cu) can react with aluminum (Al), which causes Al_2Cu during the "sinter + forging" combined process.

These analyzes indicate the advantage of forging as the second process and then the advantage of sintering during the manufacturing combined process. The homogeneous

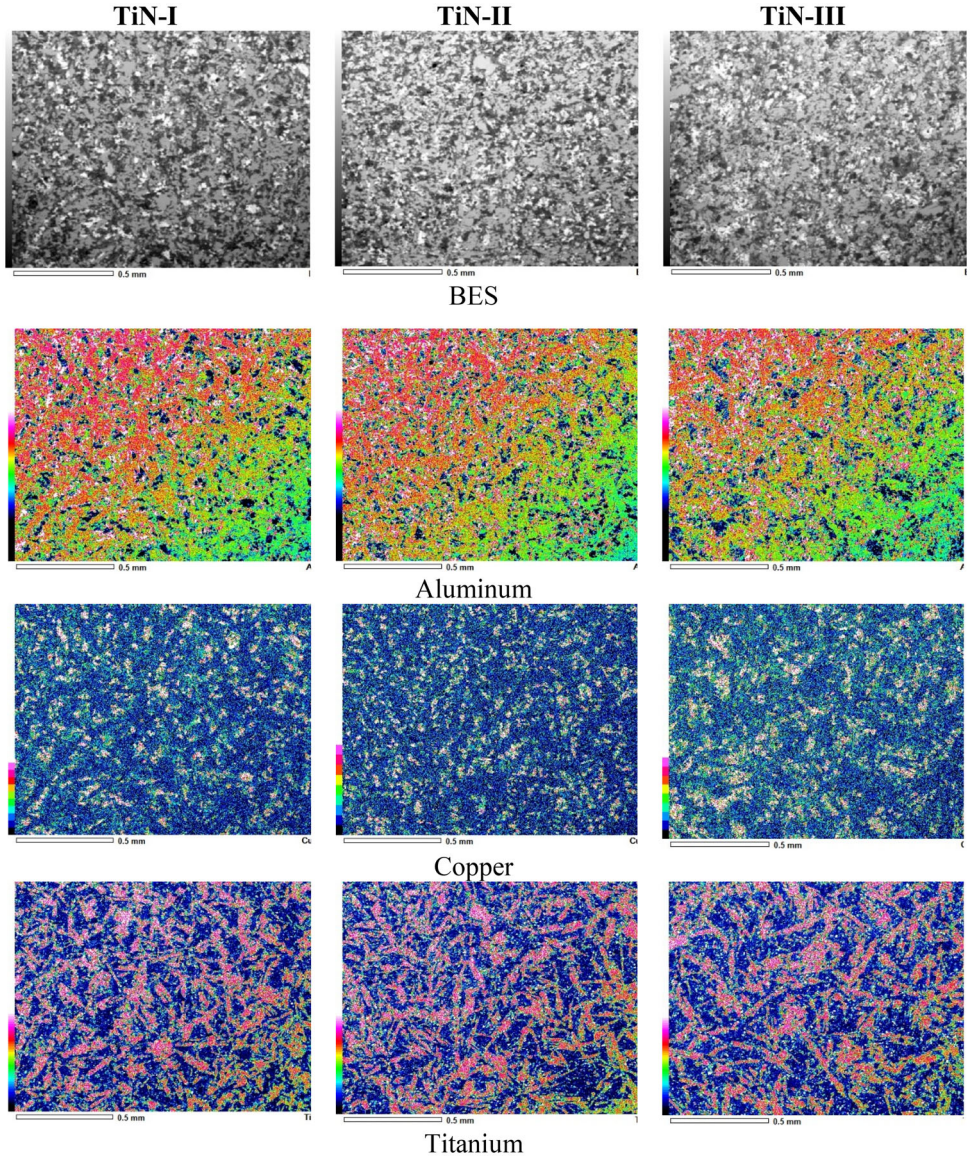


Figure 6. SEM imaging and EDX mapping for aluminum, copper and titanium coming from the reinforcements. Mapping analyses were made on the polished surfaces for three composites.

distribution of the matrix-reinforcements and the wetting facility are due to the final stage of the forging process as shown in Figure 6. It is a simple operational process to control the tight (non-porous) distribution of reinforcement elements in the matrix phase, through the combined manufacturing method proposed in the study. For this reason, a globular structure and a semi-perfect interface were obtained within the internal structures of hybrid aluminum metal matrix composite materials through an innovative combined manufacturing method. By designing and manufacturing hybrid metal matrix composites with this combined method, it provides an important advantage by providing high toughness at low cost. In the previous papers [4, 9, 15] examined within the scope of this study, the effects of AMCs reinforced with hard constituents in combined manufacturing processes on the toughening properties were examined. In addition, these effects have been determined in detail to more clearly demonstrate the benefits of the combined manufacturing method [2, 4, 5, 7–9, 13–18].

3.2. Evaluation of destructive test results

3.2.1. Wear performance test results

In order to reveal the wear performance of composite specimens manufactured within the scope of our research, scratch tests, one of the destructive test methods, provided very useful and valuable data. During this experiment period, large shear stresses occurred on the contact surface due to the friction contact between the Zirconium ball indenter and the surface of the composite specimen. While zirconium ball slides on the surface of the indenter composite specimen, it creates tangential tensile stresses on the back surface of the indenter and tangential compression stresses

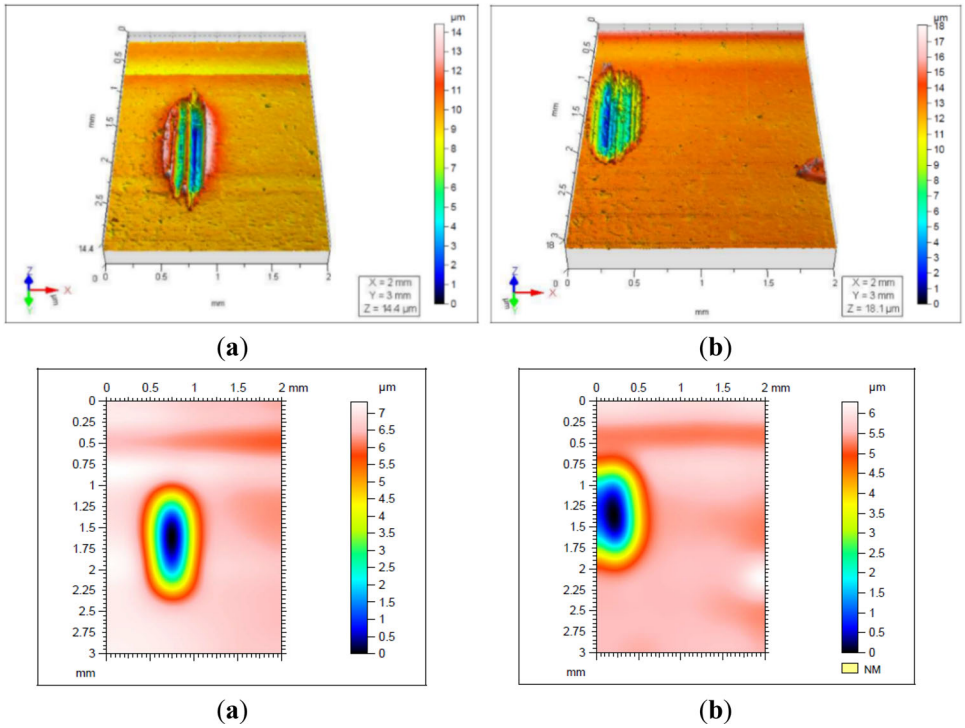


Figure 7. 3D wear damage zones of TiN-I for: (a) 50×10^3 cycles and (b) 100×10^3 cycles.

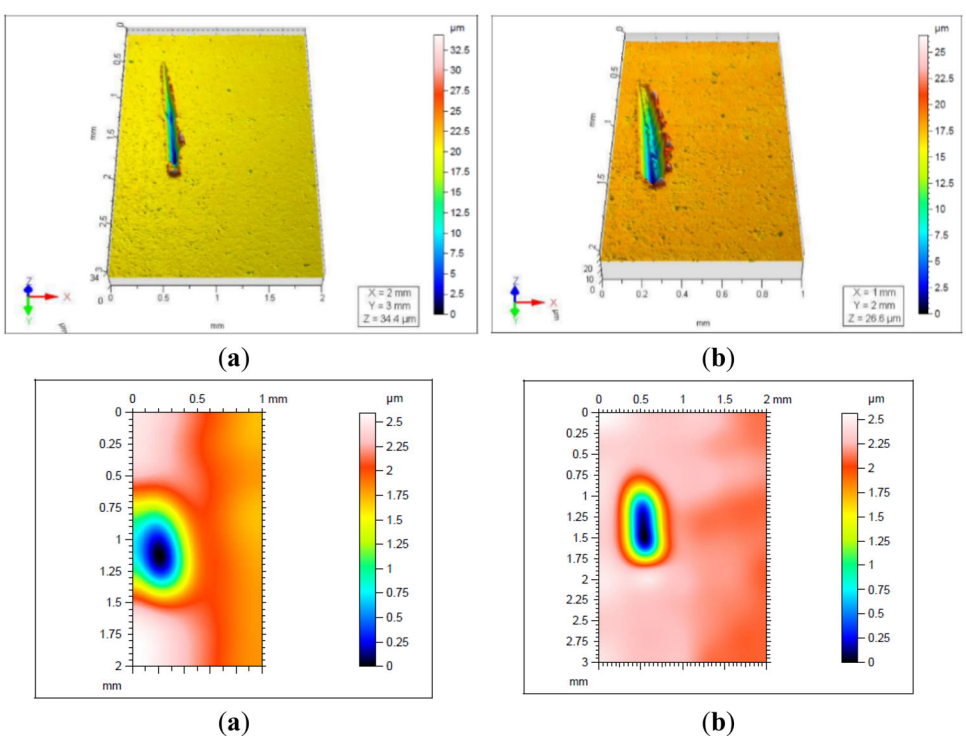


Figure 8. 3D wear damage zones of TiN-II for: (a) 50×10^3 cycles and (b) 100×10^3 cycles.

on the front surface of the indenter. As can be seen in the detailed literature studies, these high shear stresses have been determined as the most important reason that shows wear by spreading between the matrix/reinforcement of the composite specimen [2, 4, 5, 9, 13–16, 18–21, 24–26].

Wear performance of TiN-I, TiN-II and TiN-III specimens were carried out (under 1.9 N force, at 15 Hz frequency, in 50×10^3 and 100×10^3 cycle repetitions). These 3D test results were shown in Figures 7–9 (wear damage zone, characteristic surface parameters).

The results of the scratch tests are summarized in Table 2. It is evident from these results that introduction of TiN particles into the composites improved their wear resistance. The results indicate that increase in the content

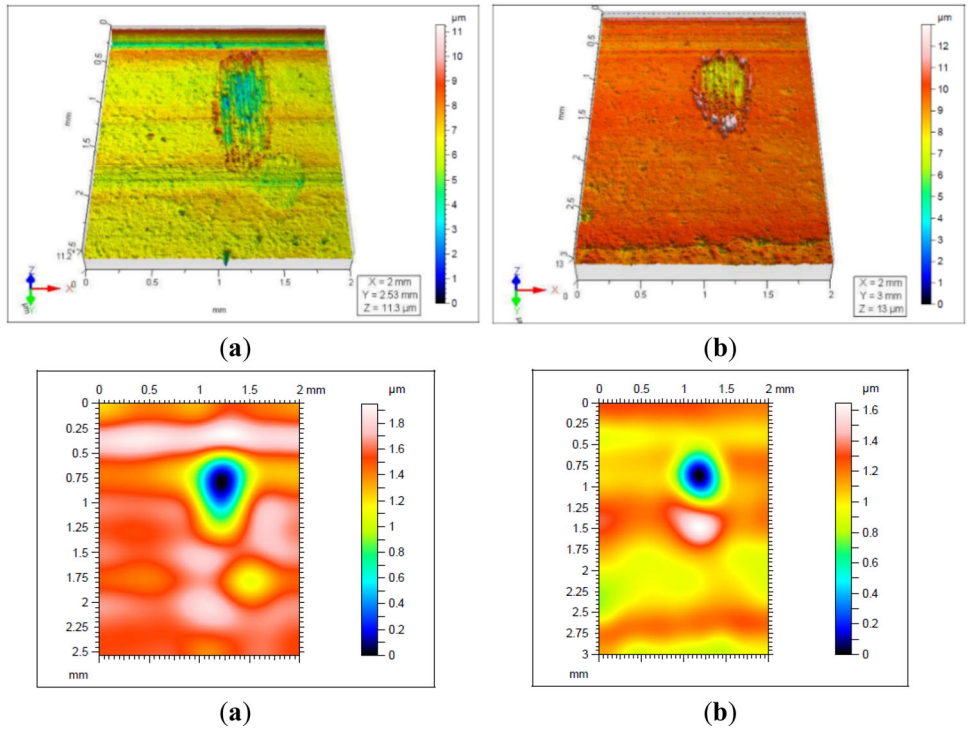


Figure 9. 3D wear damage zones of TiN-III for: (a) 50×10^3 cycles and (b) 100×10^3 cycles.

of the fine, hard TiN particles is liable for the higher wear resistance. At the beginning of the scratch test, damage mechanism is seen as adhesion type, after that the damage mechanism is mixed generally abrasive and also a little amount oxidation is observed at the final stage of the scratch. We believe that the manufacturing process; sinter + forging at the industrial scale can improve the scratch resistance behavior better than the results presented here under the laboratory scale. It means that for example, the specimens tested for longer period (100,000 cycles) containing 30% wt. of fine hard TiN particles have given very satisfactory results. One can believe that the well compacted specimens have shown very tight chemical bonding diffusion (diffusion of particles into the matrix resulting in very strong chemical bonds) between the matrix and TiN particles which should be further improved at the industrial scale. Hence the results obtained are very excellent application for the industrial pieces. Evidently, adjustment of the operational parameters of the test equipment could be improved in the laboratory scale. For this reason, these tests are going on under the industrial scale carried out by our aeronautical partners.

3.2.2. Micro hardness test results

As indicated in Sect. 3.1, the well dispersion of the reinforcements in the matrix has influenced micro hardness evolution in the structure. The values presented in Figure 10 have shown the major effect of the new process carried out in this work. These values are obtained by taking the interface between the main matrix phase and the reinforcing components. Hardness measurements of the three compositions indicate that there is a considerable increment during the applied sinter + forging process depending on the TiN content (Figure 10). This is because of an increased volume of the reinforcing hard phases (TiN) in the Al matrix that also show the improved densification of the structure. The improvement of hardness after hot pressing is because of enhanced densification. The process should improve the toughening of the composites due to the homogenous dispersion of the reinforcements in the structure.

3.2.3. Quasi static compression test results

This new composite design thinking (sinter + forging) developed within the scope of the study and laboratory scale can be developed as a larger scale and economical method for the manufacturing of parts belonging to different types of industry. Generally, using aluminum powders as a matrix, microstructure can be developed very well with this method. Sinter + forging process, also called a new manufacturing method, provides high dimensional precision in the manufacturing of industrial parts [13, 14, 16, 17]. For this reason, this method is used for bulk materials in industrial applications. Parts manufactured with low cost sinter + forging manufacturing method (in many different industry areas) show high performance (compression, bending, impact, toughness, wear, hardness, fatigue, etc.) in many different ways against challenging service conditions. Although it is found only in industrial parts manufactured by sintering (high temperature, low pressure) or only forging (low temperature, high pressure) in the industry, manufacturing (optimized temperature and pressure) with sinter + forging method, which contains the superior features of both

Table 2. Defining of damage results with a scratch test for two different cycles.

Type of materials	Damage in surface area and volume calculated via software	No. of cycle 50×10^3	No. of cycle 100×10^3
TiN-I	Surface (mm^2)	0.440	0.493
	Volume (μm^3)	2,379,275	2,379,560
	Depth-Height max. (μm)	14.5	14.6
	Depth-Height mean (μm)	5.40	5.61
TiN-II	Surface (mm^2)	0.433	0.486
	Volume (μm^3)	899,974	920,984
	Depth-Height max. (μm)	24.3	25.2
	Depth-Height mean (μm)	6.28	7.21
TiN-III	Surface (mm^2)	0.412	0.473
	Volume (μm^3)	526,530	528,964
	Depth-Height max. (μm)	5.65	5.72
	Depth-Height mean (μm)	5.65	5.79

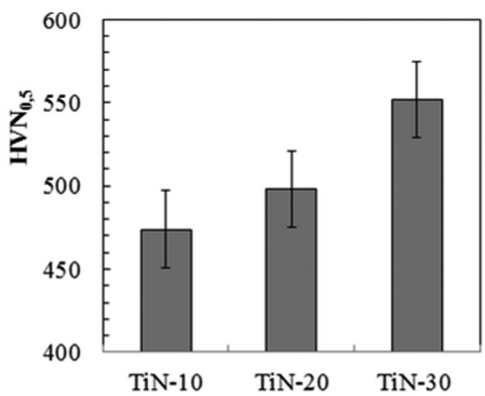


Figure 10. Micro hardness evolution of the three composites, TiN-I, TiN-II and TiN-III respectively.

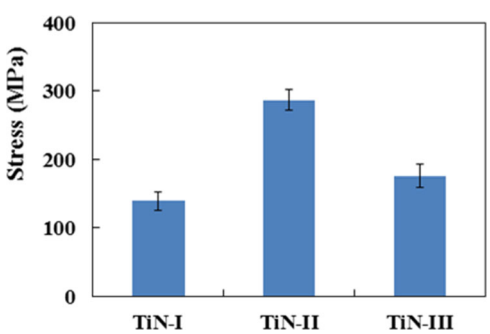


Figure 11. Quasi static compression test results for three composites (max. stress capacities), TiN-I, TiN-II and TiN-III respectively.

manufacturing methods, it can be made to be higher performance with lower cost. This situation briefly means the following; with this manufacturing method (sinter + forging), tougher and higher strength parts can be obtained at lower cost than other manufacturing methods. Quasi-static compression test results, which are one of the mechanical behaviors of innovative composite specimens manufactured in our research, are shown in Figure 11.

Fracture surfaces of these composites have been shown in Figure 12. In observations made on all fractured surfaces of three hybrid metal matrix composite specimens with different compositions, it has been demonstrated that a tight chemical bond is formed between the reinforcing components and the main matrix phase by diffusion mechanism. The reinforcements play a good bridge between two parts in the matrix thanks to a good cohesion at the interface between the matrix and the reinforcements.

It is interesting thing to explain here that the fracture initiation site and fracture propagation lines are always found in the intermetallic phase, Al_2Cu site not the interphase at the high fracture strength level. There are any cracks and/or secondary cracks found at the interphase between the aluminum matrix and the secondary phase, TiN. However, this present research is going on with some real aeronautical and aerospace engineering applications and some other results that were not shown here really worth to be discuss because the surfaces of the TiN hard particles were really activated with well wetting capacity and attached very well to the matrix but it stimulates for creating tight intermetallic phases in the composition during sinter + forging process.

3.2.4. Three point bending (3PB) test results

It is a multifunctional manufacturing method that can include many functions such as the main target underlying the innovative composite design reached in the last of the detailed literature research, providing low production cost on an industrial scale, easy adaptability to the industry, full and high performance of the parts produced, and precision dimensional accuracy of the parts [2–5, 13, 14, 16–18].

Within the scope of our research, 3-point bending test, which is one of the destructive test types, was performed and the effect of the combined sinter + forging manufacturing method and other components in the composite internal structure, which is an innovative manufacturing method, was analyzed on the developed composite specimens. Figure 13 shows these 3PB test results for experiments obtained on the three types of composites. In addition, fracture surfaces of these for three composites for the TiN-I, TiN-II and TiN-III were illustrated in Figure 14. The presence of a high reinforcing element, we can observe that it constantly provides higher resistance under 3PB conditions. The low reinforced (10% TiN) composition appears to be more ductile in bending. However, brittleness was observed in some of the specimens produced for the third composites (30% TiN) and some of the specimens have fractured early as indicated in Figure 13, but they were not generated in regular way. It seems that this is caused by the operational parameters. For all that, some γ-Al_2O_3 particles are good homogeneous, even if the hard particles from the distribution of the 3PB test

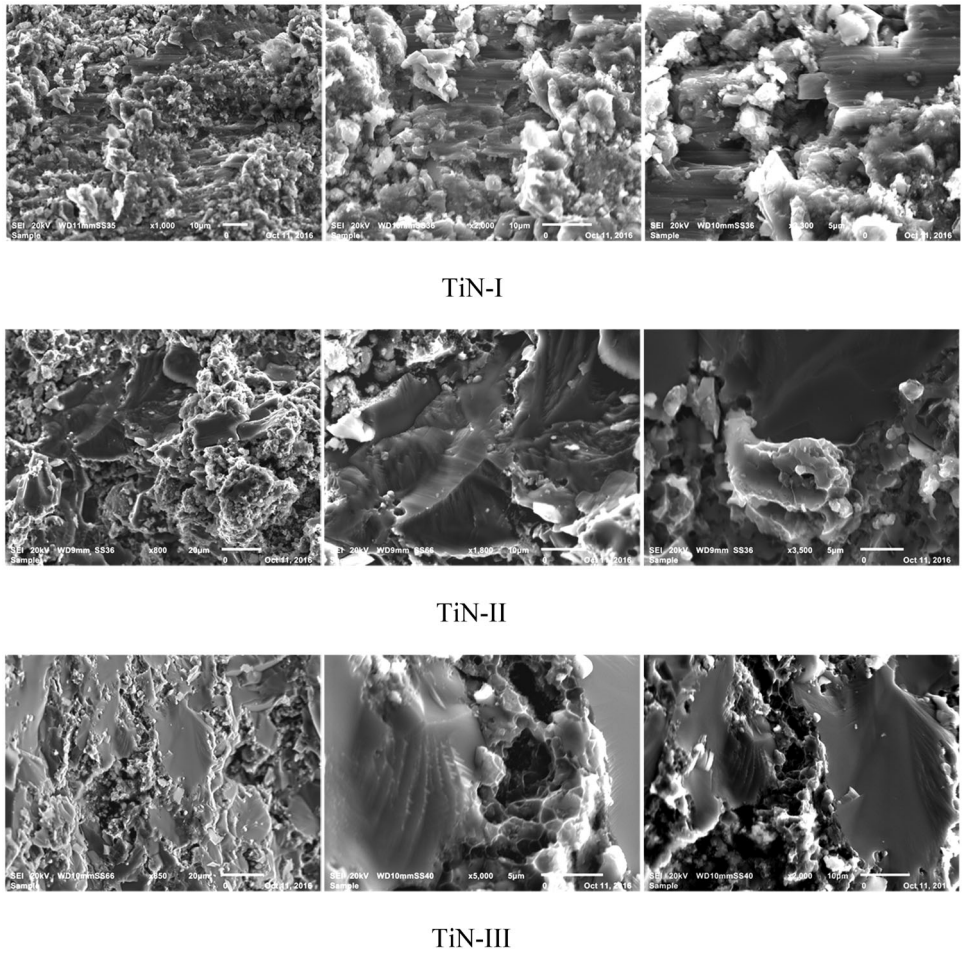


Figure 12. Fracture surfaces of these for three composites for the low (left column) and high (middle and right column) magnification for the TiN-I, TiN-II and TiN-III respectively.

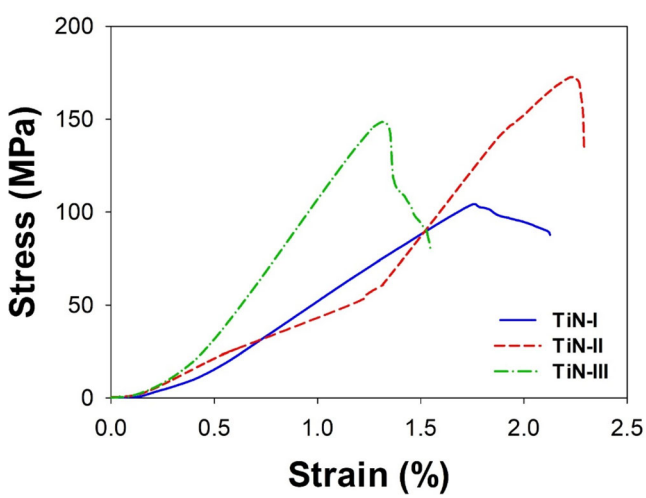


Figure 13. 3P Bending Stress-Strain test results for three composites containing 10, 20 and 30% TiN respectively.

specimens are clustered in the structure strictly linked to the milling conditions. These problems come from the laboratory (operational) conditions and evidently can be improved the production of the pieces under much better conditions in the industrial scale for the real aeronautical and aerospatial applications. Here we try to give a useful tool and idea for manufacturing engineers for the production with low-cost high-performance composites.

4. Conclusions

A novel design of the aluminum matrix composites reinforced with hard particles (TiN and γ-Al_2O_3) performed through the combined method (Sintering + Forging) of powder metallurgy for substantial densification in order to expand mechanical essentially toughening properties. This present project is going on with aeronautical/aerospatial industrial partners and some preliminary results could be driven from the results that have been presented in the frame of this research program.

In the microstructural analysis carried out on composite specimens developed and manufactured by the combined sinter + forging manufacturing method within the scope of our study, it was determined that there was non-porous and a strong chemical bond diffusion especially in matrix/reinforcement interfaces. Doping and preparation conditions should be further improved in order to improve wear and ductility behavior, which are mechanical properties of these composite structures. Ball milling is required over a longer period of time for the reinforcements in the matrix phase of

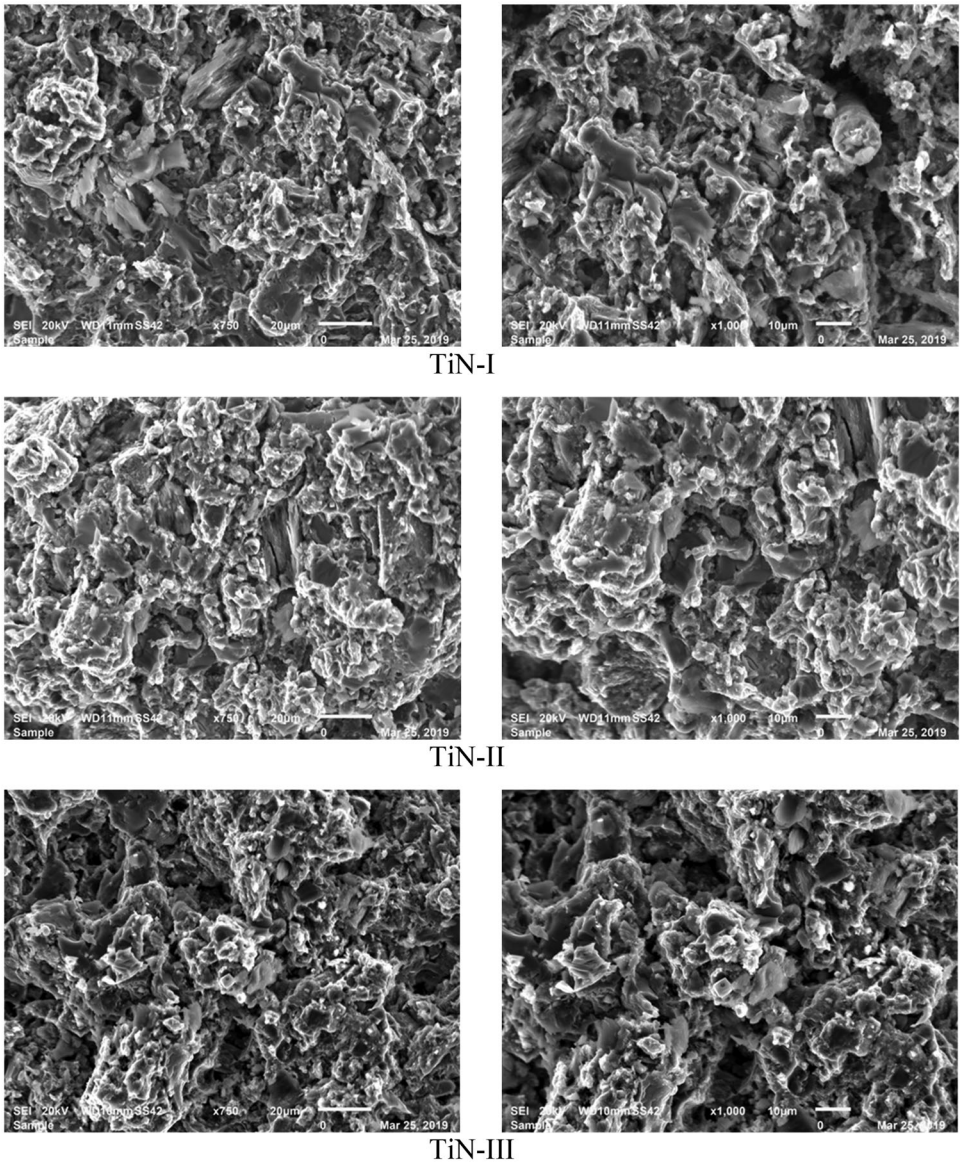


Figure 14. Fracture surfaces of these for three composites for the low (left column) and high (middle and right column) magnification for the TiN-I, TiN-II and TiN-III respectively.

the composite material to be further reduced in size, to be geometrically similar to each other and to have a higher homogeneous distribution.

The structural behavior of composite materials developed within the scope of our research is more advantageous than composite materials developed by other manufacturing processes such as melting, reactive meta infiltration and others. In order to manufacture parts on an industrial area and to optimize the parameters such as machining parameters and reinforcement content rates during the manufacturing process, it is necessary to conduct more extensive experimental research. Within the scope of the study, it was aimed to better understand the effect of reinforcements on the optimization of toughening properties of the aluminum-based composites. Therefore, important indicator parameters on this feature have been determined by certain tests performed at room temperature.

Acknowledgements

This work has been performed partially in the frame of research project for aeronautical/aerospace applications. Authors acknowledge and appreciate so much to Dr. H-A. ALHAS from Airbus Space and Defense in London/UK for supplying materials and for technical help. We also acknowledge to the electro-chemistry department of CNAM-Arts et Métiers for the chemical treatment (wettability-surface activation for the powders specially for TiN).

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