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Incorporation of SiC Ceramic Nanoparticles into the Aluminum Matrix by a Novel Method: Production of a Metal Matrix Composite

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Abstract

SiC ceramic nanoparticles were incorporated into the A356 aluminum matrix with different compositions using a combination of stir casting and semisolid extrusion. The microstructure and mechanical properties of the produced nanocomposites were evaluated. The results showed that the presence of Nickel acts as an appropriate metallic carrier for SiC nanoparticles, which causes uniform dispersion and spherical grains. Consequently, the coexistence of SiC nanoparticles and Nickel resulted in UTS of above 304 MPa and elongation of 5.8%. However, the addition of Titanium caused the formation of flake-like intermetallics, which decreased the elongation of the nanocomposites. The method introduced in this study for the incorporation of SiC ceramic nanoparticles can be used as a promising process instead of conventional methods, which are expensive and time-consuming.

Keywords SiC · Ceramic nanoparticles · Nanocomposite · Aluminum

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Introduction

Aluminum matrix nano-composites (AMNCs) are used because of cost-effective manufacturing methods and attractive mechanical properties in a variety of industries, including aerospace, automotive, and sports [1,2,3,4,5,6,7,8,9]. The main goal of AMNCs research is to use ceramic nanoparticles to improve ductility [5, 10,11,12,13,14,15]. AMNCs are generally produced by powder metallurgy [16,17,18] or conventional stir mixing [19,20,21,22,23], while both the methods have some drawbacks. Powder metallurgy is costly, while conventional stir mixing followed by casting is one of the most economical techniques available to produce large near-net-shape parts from metal matrix composites and a good deal simpler than powder metallurgy, but the poor wettability of ceramics by molten metal makes it challenging to incorporate ceramics into molten metal and then homogeneously disperse them. For example, the floating and settling of nanoparticle clusters have been observed and reported by researchers. Another disadvantage of conventional stir mixing followed by casting is that the morphology of the eutectic region after solidification, especially in aluminum–silicon alloys, is generally non-optimal. Due to the eutectic reaction principles, the Si particles appear in the form of coarse sharp needles and/or platelets that mainly affects the mechanical properties. Therefore, their microstructural modification has been reported several times [24, 25] by using other thermomechanical steps like extrusion after casting. Although thermomechanical processing through repetitive hot/warm rolling/extrusion of as-cast products can produce well-refined microstructures, it is extremely costly. It harms the anisotropy and ductility as a strong basal texture develops.

During the last two decades, different researches have been performed on semisolid metal (SSM) processing [26, 27] with some benefits such as reduced porosity and solidification shrinkage, and therefore improved mechanical properties. The mixing of the reinforcing phases in the semisolid state can influence breaking the gas layer due to a high melt viscosity of the melt. In our previous studies [28], to increase the wettability of nanoparticles with the melt of A356 alloy, solid-state modification of ceramic nanoparticles in a ball-mill was conducted, and composite powders with superstructures were injected into the semisolid melt. It was found that stirring in the viscous melt was essential but not enough, and it seems that liquid-state stirring is also required for more composite powder breaking.

In this study, using a three-step process including semisolid stir casting, liquid stir casting, and twin-screw SSM process [29], as a complementary casting process, was implemented to improve the properties of A356/SiC nanocomposites and reduce the traditional casting defects for AMNCs.

Experimental Procedures

Materials

A356 aluminum ingot containing 7.2 wt% Si, 0.33 wt% Mg, and 0.11% Fe was used as the matrix. 1 wt% of SiC ceramic nanoparticles with a size of 70 nm were used as the reinforcement phase. Nickel and Titanium were used as the modifying agent of nanoparticles in a ball mill to aid better incorporation into the semisolid melt. Further details about the nanoparticles and carrier agents have been reported in our previous work [30].

Processing

Details of the ball-milling process as well as composite powders preheating before casting have been reported elsewhere [13, 30, 31]. As mentioned, a hybrid casting process was used in this study. The first step (semisolid stirring) was designed to physically entrap the reinforcement material that was in modified form after the ball-milling process. The details of the casting procedure in the semi-solid state were reported in our previous studies. The second step (liquid-state stirring) was designed to improve the breaking of composite powders, which were mechanically stirred in the semisolid state with a high level of shearing for releasing the nanoparticles by the shear stress generated in the semisolid melt. In

this step, stirring was stopped after 6 min of stirring in the semisolid condition. Using a 25 °C/min rate, the temperature was increased up to 680 °C. Then, the melt was stirred at this temperature for an extra 2 min to achieve a higher release of the nanoparticles. After the completion of the stir casting process, the bottom valve was opened, and the melt was guided to a twin-screw semisolid extrusion setup. The nanocomposite slurry conveyed forward while it was sheared at a solid fraction of 0.3 by two intermeshed-self wipe screws. These screws co-rotated and pressurized slurry to the die assembly to form an extruded rod of the alloy. The processing time and die orifice diameter were 25 s and 10 mm, respectively.

Tensile behavior of the rolled specimens with a plate shape and a gauge length of 8.3 mm was done based on the ASTM E8M standard three times utilizing a DY-28 testing machine with an initial strain rate of $2 \times 10^{-3} \text{ s}^{-1}$ at room temperature. The microstructure of the nanocomposites was investigated using optical microscope (OM) and field-emission gun scanning electron microscopies (FESEM) equipped with energy dispersive spectroscopy (EDS).

Results and Discussion

The microstructure of the A356 aluminum ingots is shown in Fig. 1, which shows that the microstructure included α -Al phase and eutectic Si phase. As mentioned, Ni and Ti powders were used to modify the nanoparticles for avoiding floating or settling of nanoparticles, and composite powders were injected in the semisolid melt followed by the liquid stirring and finally semisolid extrusion. The OM and FESEM microstructures of the Ti-SiC and Ni-SiC prepared reinforced A356 samples are shown in Figs. 2 and 3, respectively.

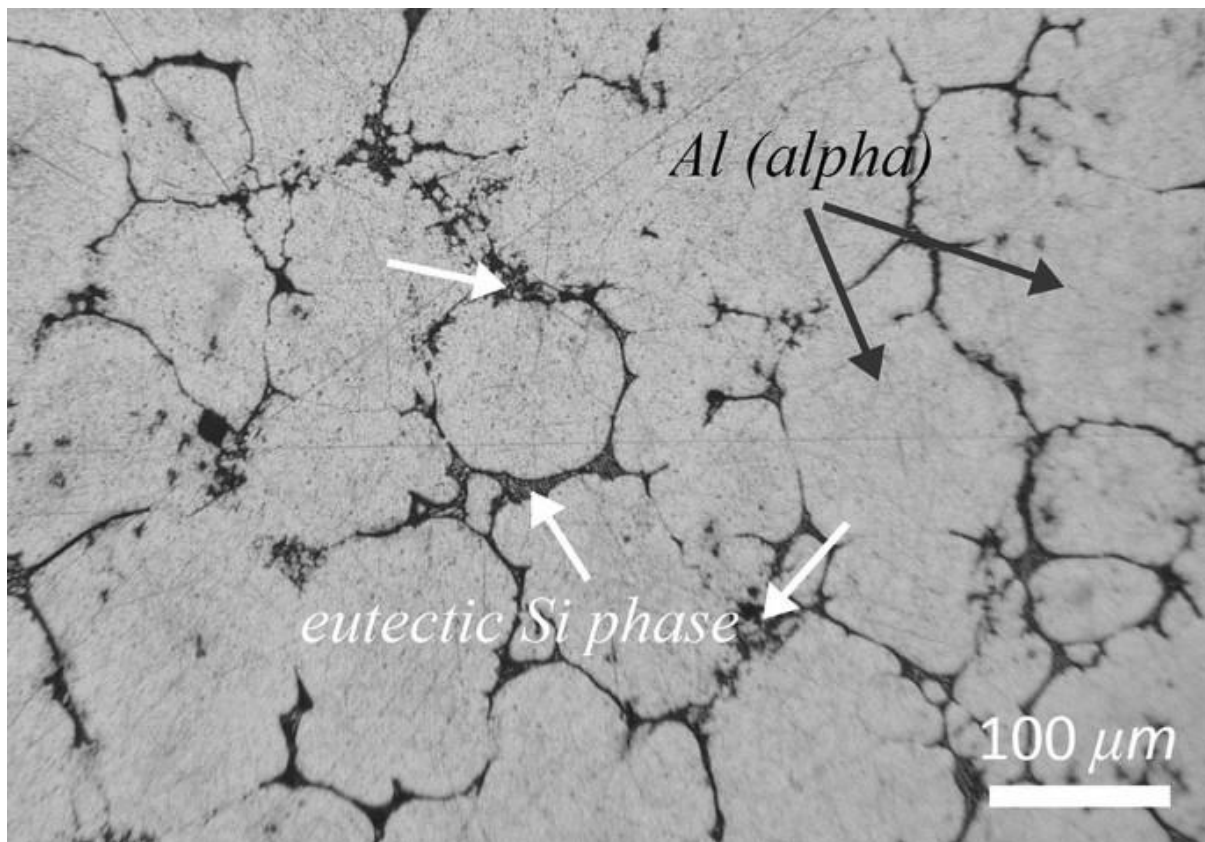


Fig.1. The microstructure of the A356 aluminum ingots

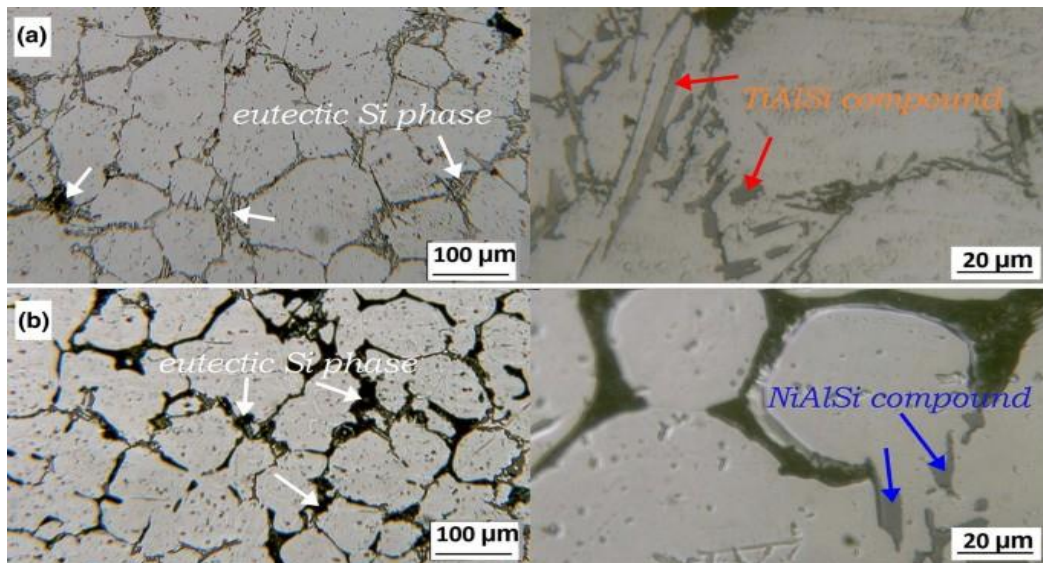


Fig.2. The OM images of A356 reinforced with a Ti-SiC and b Ni-SiC nanocomposites after semisolid extrusion at low and high magnifications

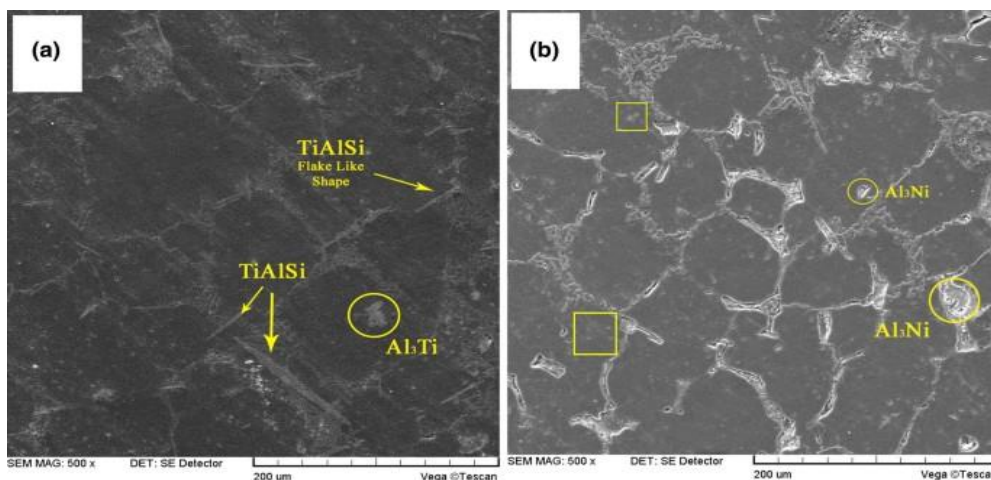


Fig.3. SEM images of A356 reinforced a Ti-SiC and b Ni-SiC nanocomposite samples after semisolid extrusion at low and high magnifications. (Color figure online)

Figure 2a shows the OM images of the Ti-SiC sample with low and high magnifications. The α -Al phase was detected with a tendency for spheroidization of the eutectic phase in some zones. The TiAlSi compound with a flake-like morphology can also be observed around the eutectic Si phase at higher-magnification, similar to what was previously found when Ti is added to A356 melt [32]. Some fragmentation of the eutectic Si phase is also visible in some areas after extrusion due to the high shear forces generated in the semisolid state.

Figure 2b shows a different microstructure of a nanocomposite containing nickel as a modifier. The more spherical α -Al phase was detected that was surrounded by a network of nickel-rich phases and modified eutectic silicon phase, and lower grain size was obtained for this sample. Figure 3 shows the SEM images of these two samples, and some traces of sub-micron pores can also be seen, indicating that the semisolid extrusion could not completely remove all porosities. Also, it seems that the presence of Nickel and Titanium as carrier and modifier agents of SiC nanoparticles was highly active in control of the microstructure of nanocomposites. It seems that the interaction of Nickel with the molten aluminum at 680 °C will lead to the formation of the Al₃Ni intermetallic phase [33] and will have some

effects on the grain growth and morphology of the eutectic silicon as these intermetallic phases are formed around the eutectic silicon at the grain-boundaries. It should be noted that Nickel and Titanium have a high Hamaker constant between 350 and 450 zJ [34], and their presence will be highly useful for the capture of nanoparticles during solidification of aluminum melt when a core-shell structure of Nickel-SiC and Titanium-SiC is formed. Some researchers [35, 36] have used metallic carrier agents for the nano ceramic particles and reported that the carrier agent not only causes injection of nanoparticles into the melt and avoids their interaction with the alumina surface layer, but also it will affect the type of interaction between nanoparticles and solidification front. While it seems that they must keep their core-shell structure after stirring in the semisolid and liquid melt to be effective for complete integration with the metal after solidification.

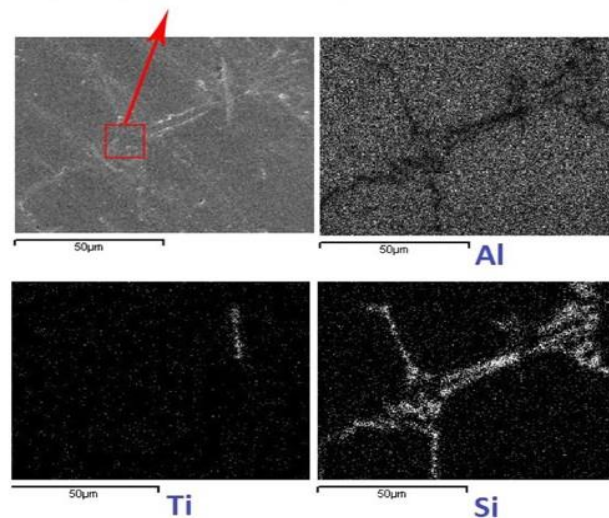
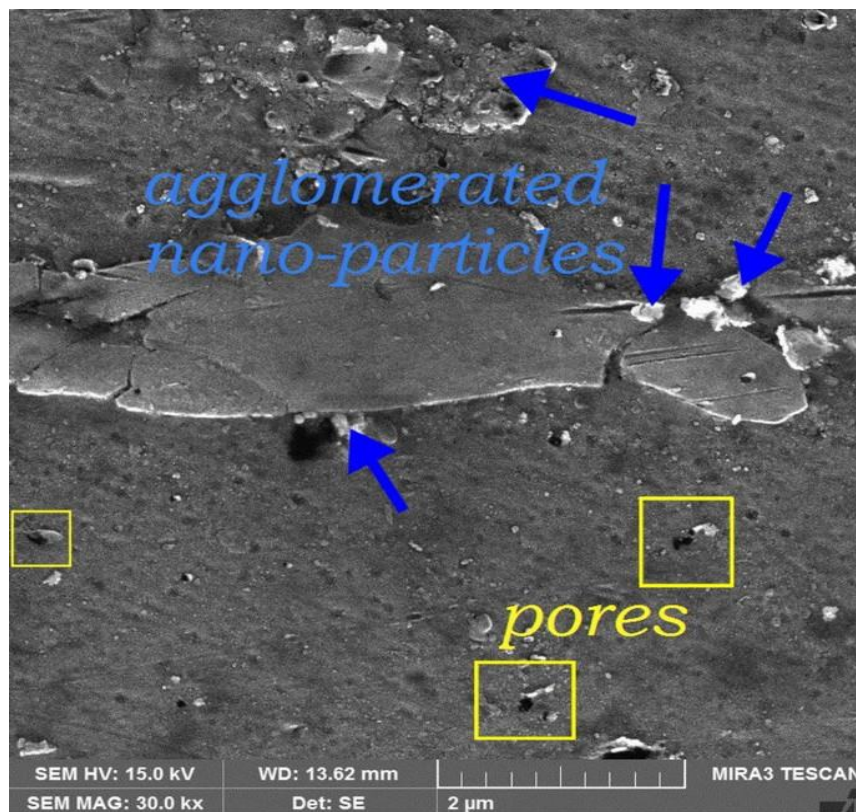


Fig.4. EDS images of Ti-SiC nanocomposite after extrusion. (Color figure online)

Previous reports [34] indicated that Nickel and Titanium have a limited solubility in aluminum. Instead, they have an extreme tendency for an exothermic reaction with molten aluminum to form intermediate phases and release the heat and our previous work [28] indicated that this heat is very effective on the releasing of nanoparticles into the melt during the stirring process, and therefore, the nanoparticle shell is expected to be dissolved by the continuous stirring and the Nickel and Titanium core will also interact with the melt so they might not be effective at incorporating the nanoparticle against the solidification front.

Figures 4 and 5 show the FESEM/EDS of the samples with higher magnification to show the nanoparticle presence in the matrix of aluminum and discuss the solidification in these samples. EDS map results in Fig. 4 shows the detection and presence of silicon and Ti-rich intermetallic phases at the grain boundaries and in a high magnification FESEM image, with a proper distribution of single nanoparticles. Some agglomerated nanoparticles can be observed around a eutectic silicon phase, while this suitable dispersion was not found in the areas far from grain-boundaries (middle of grains). Also, sub-micron sized pores, highlighted with the yellow-colored rectangles that remained after extrusion.

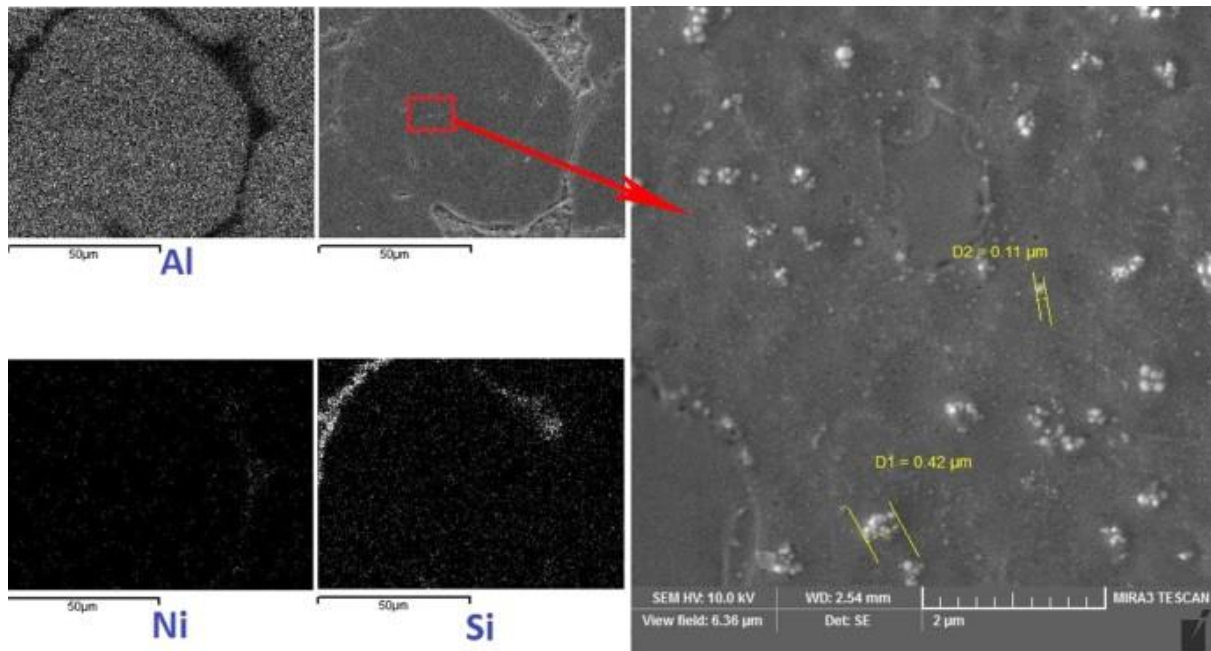


Fig. 5. EDS images of Ni–SiC nanocomposite samples after extrusion. (Color figure online)

Figure 5 shows the FESEM/EDAX microstructure of the Ni–SiC sample, in which nickel-rich phases at the grain boundaries can be observed. However, in contrast to the previous sample, nanoparticles were readily detected in the middle of α -Al grains, showing the engulfment of nanoparticles in front of ahead of the solidification facade, which causes the joining of nanoparticles. Ferguson et al. [37] indicated that ultrafine nanoparticles were engulfed ahead of the solidification front in low concentrations.

If the nanoparticles can pass the oxide film and enter to the melt, then they have a high chance for capturing as their concentration in this study is 1 wt%. However, they indicated that sub-micron ceramic clusters or particles above 100 nm diameter size might have a lower chance of engulfment. Figure 5 counters this matter in that some ceramic particles above 200 nm were also captured by the solidification front.

Figure 6 shows the schematic of the obtained microstructures after extrusion, which was produced according to the data of current study and experimental results of other researchers [38, 39]. It can be observed that carrier agents (red-colored parts) modified the nanoparticles, and deagglomerate them during intensive collisions of balls in the ball-mill, and some superstructures were obtained before casting. Then nanoparticles and carriers started to be released and interacted with the melt during stirring, while the solid-state modification process could not completely deagglomerate as-received nanoparticles as some clusters were still detected. Then, Al nucleus was formed and for Nickel (image No. 4 in Fig. 6) was obtained with a more suitable dispersion of nanoparticles inside the grains, while for titanium (image No. 5 in Fig. 6) was formed, in which flake-like intermediate Ti-rich phases, as well as suitable dispersion of nanoparticles around them, were detected.

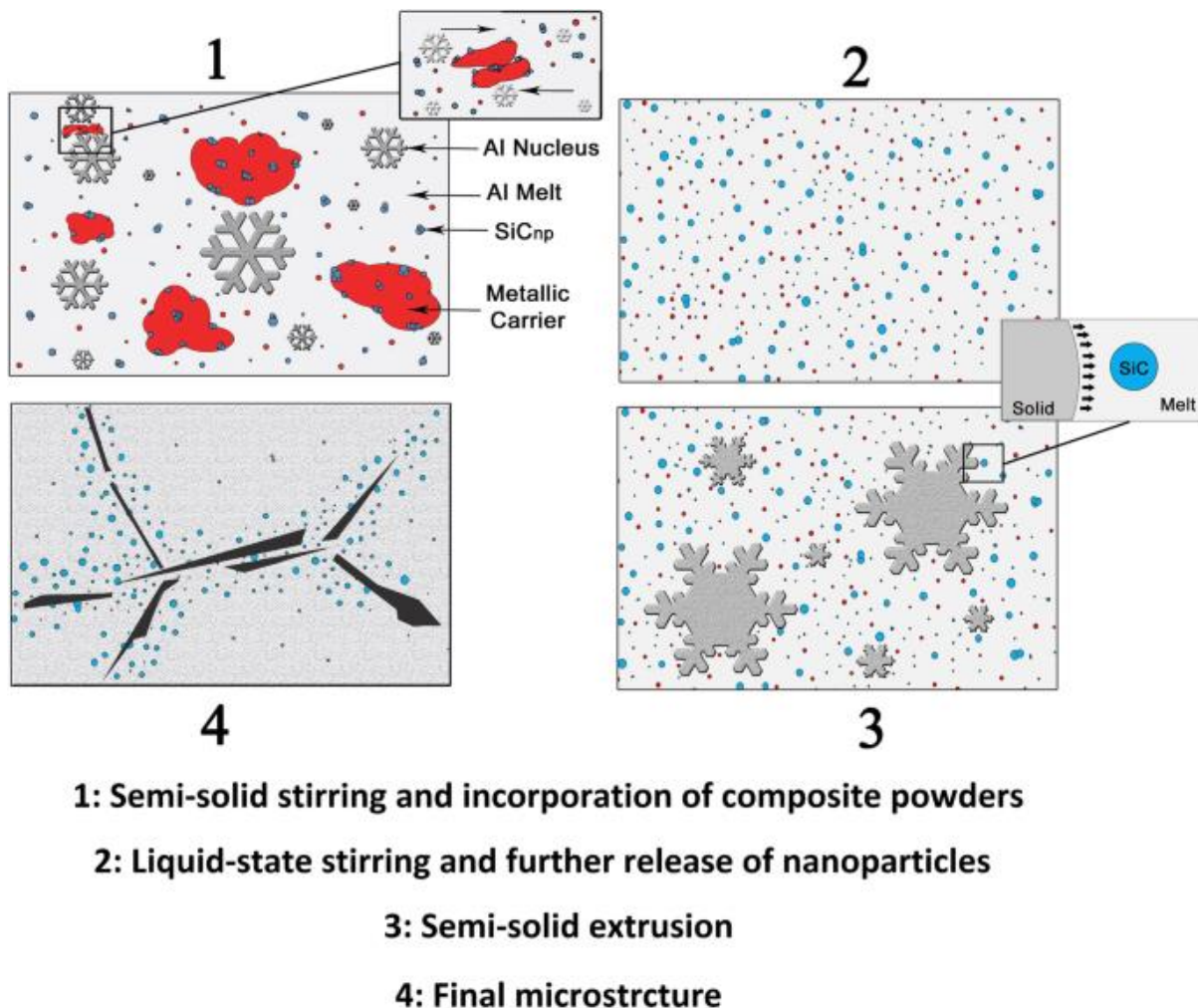


Fig.6. Schematic of solidification microstructures obtained in this study. (Color figure online)

It has been known that the agglomeration of nanoparticles is closely related to their high surface free energy densities [40, 41]. However, in this study, the addition of Ni and Ti could not help to lower the surface energy density of SiC nanoparticles so that they can be well dispersed. The mechanism of effect of Ni and Ti can be summarized as follows. The ball milling was used to produce the composite powders. By this method, first, nanoparticles will be separated from each other and a metallic layer will cover them. So, they will have a different composition at their surface with a much similar thermodynamic character to the molten metal. Second, during incorporation of nano-particles into the vortex, no direct contact will occur between separated nanoparticles with the melt. When, they transfer under the melt surface, then Ni and Ti gradually react with the melt and a suitable contact is formed

between them, and the solidified matrix is in an ideal condition. However, still some agglomeration of nano-particles can be detected after solidification showing that this process is not ideal and it can just decrease the number of agglomeration by optimizing the process parameters.

The mechanical properties of nanocomposites and as-received ingot of A356 were compared to show the total effects of 3 wt% carrier agents, 1 wt% nano SiC particles, and semisolid extrusion. From the microstructural point of view, more spherical grains were obtained for Ni–SiC nanocomposite, and for both the samples, Ni-rich and Ti-rich intermetallic phases were obtained, which for the morphology of TiAlSi phase was flake-like possibly with a high-stress concentration during the tensile test. Also, it was found that nanoparticles had a suitable dispersion even in the middle of grains for Ni–SiC nanocomposite, although some re-joining of single nanoparticles during solidification occurred. Figures 7 and 8, as well as Table 1, show the mechanical properties that were obtained in this study, and they were compared with previous results that have been reported by the other researchers, which reveals promising results. Furthermore, it was declared that the Ni–SiC nanocomposite had higher ultimate tensile strength (UTS), yield strength (YS), and, more importantly, ductility than the Ti–SiC nanocomposite. The fracture surface of the Ni–SiC sample shows a dendrite structure based on microstructural characterization. It is important to note that more than 9% ductility for A356 was reported in our previous study [22] after stir-casting followed by hot-rolling, in which severe eutectic Si fragmentation occurred, while in this study without having a secondary thermomechanical process that is expensive, around 6% ductility was obtained. By comparing the obtained results with literature, it can be concluded that using semisolid extrusion led to a combination of ductility and strength improvement in A356 based composites.

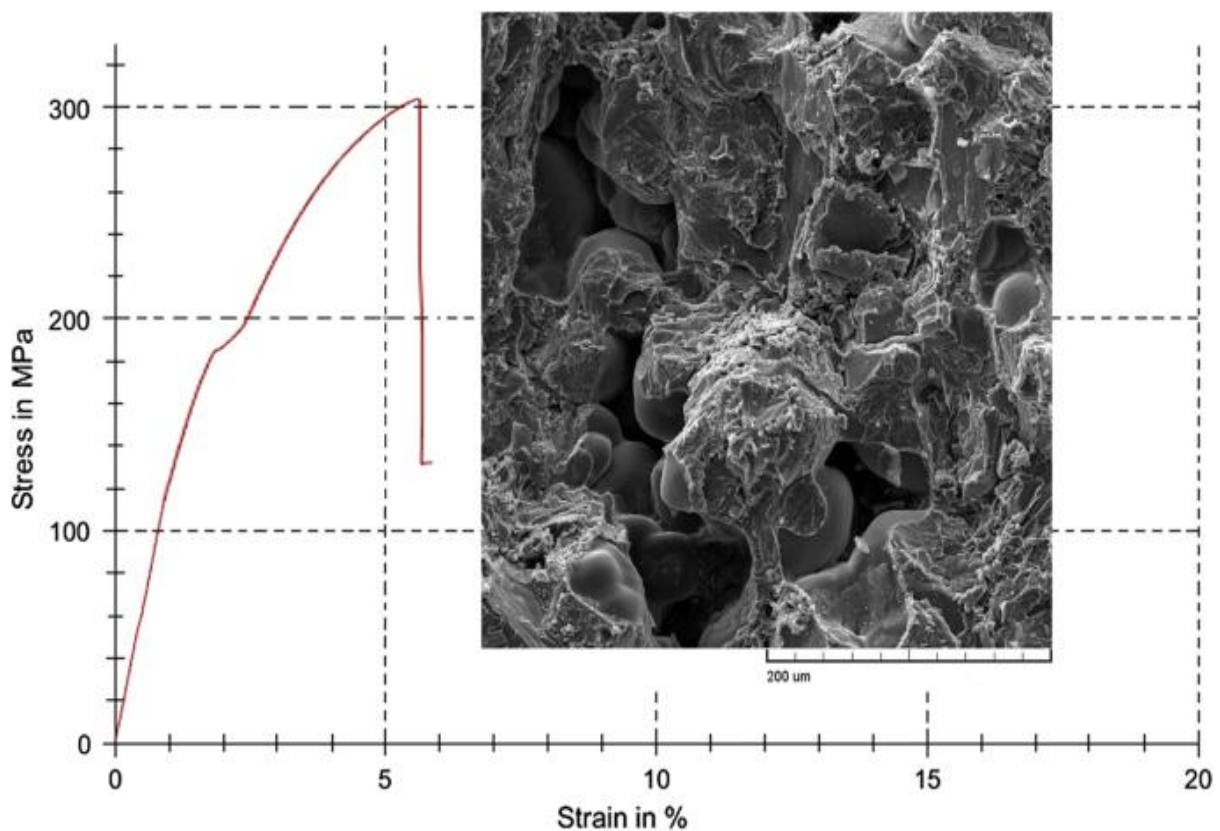


Fig.7. Stress-strain curves performed at the room temperature along with fracture surface for the Ni–SiC reinforced A356 sample

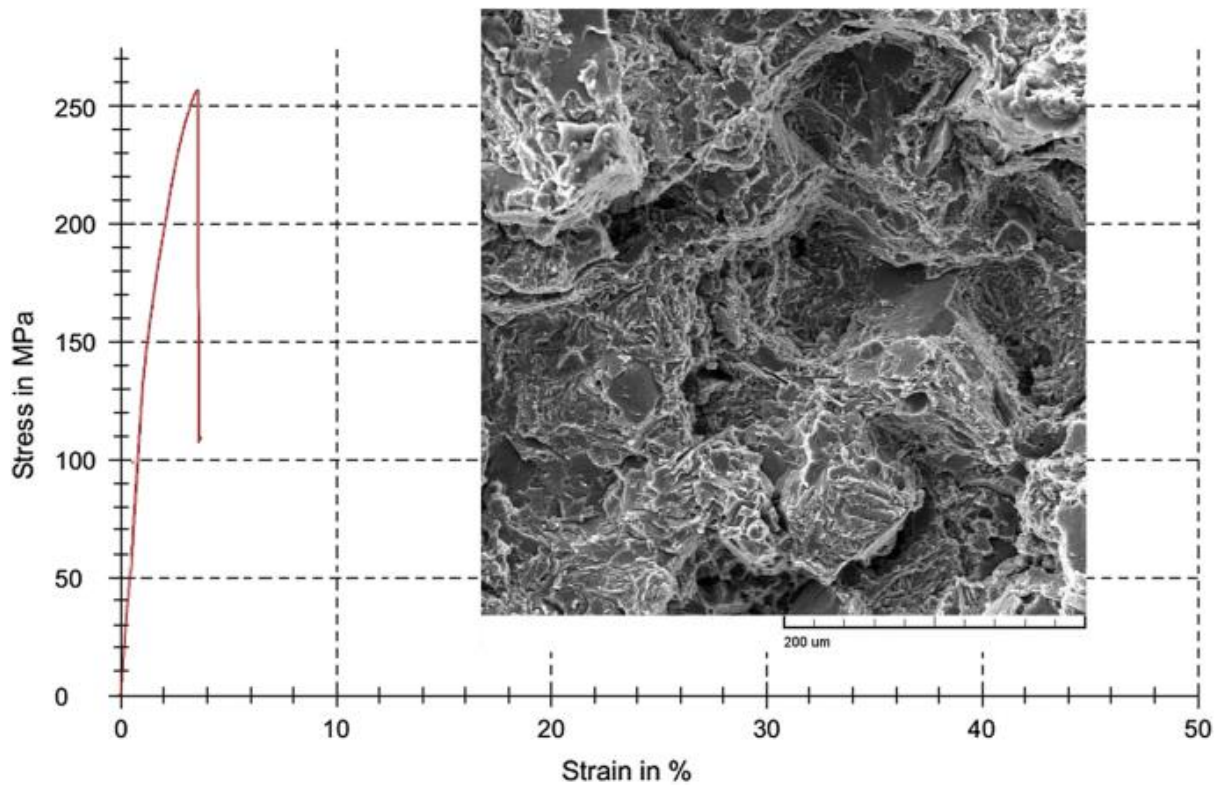


Fig. 8. Tensile behavior along with fracture surface for the Titanium-rich sample

Table 1 The mechanical properties of the samples of this study compared with literature

Sample	Process	UTS (MPa)	Strain (%)	YS (MPa)	Average hardness (HV)	References
A356	As-received ingot	146 ⁺² ₋₃	2.2 ^{+0.2} _{-0.3}	92 ⁺⁴ ₋₂	75	This study
A356-(Ti + SiC _{np})	3-steps	257 ⁺⁵ ₋₆	3.85 ^{+0.4} _{-0.6}	135 ⁺⁵ ₋₃	78	This study
A356-(Ni + SiC _{np})	3-steps	304 ⁺⁸ ₋₇	5.58 ^{+0.7} _{-0.6}	183 ⁺⁵ ₋₇	84	This study
Al356/5 vol% SiC _p	Stir casting	160	4	120	–	[42]
A356/1.5 vol% Al ₂ O _{3np}	Stir casting	265	1.12	230	100	[43]
A356/1.5 vol% TiB _{2np}	Stir casting	364	2.6	277	–	[44]
A356/1.5 vol% Al ₂ O _{3np}	Stir casting	182	2.2	115	72	[45]
A356–1 wt% SiC _{np}	Stir casting	173	5.38	137	–	[46]
A356-5 wt% Al ₂ O _{3np}	Stir casting	186	1.27	135	85.7	[12]

A higher toughness can be obtained by using semi-solid extrusion compared with stir (vortex) casting thanks to further fracture of eutectic silicon and elimination of porosities. In future works, by having a focus on higher solid fractions during extrusion, we will hope to have finer microstructure. Also, lower than 1 wt% nanoparticles as well as 3 wt% of carrier agents will be examined. It is notable that in a nanoparticle-reinforced composite, the interface effect always plays an important role, which leads to a size-dependent behavior of mechanical properties [47,48,49]. In the current research, nanoparticles with a given size of 70 nm are used. Thus, the UTS and elongation of the composites will change if the nanoparticle size changes, and future works are needed to disclose this effect.

Conclusions

SiC ceramic nanoparticles were inserted into the A356 matrix to produce nanocomposites using a novel hybrid casting technique by the combination of stir casting and semisolid extrusion. The following findings can be summarized. Using the presented innovative hybrid casting technique results in a desirable distribution of SiC nanoparticles, which does not need a secondary thermomechanical process. Thus, the presented method is more economical than the conventional methods. Ni performances as a suitable metallic carrier for SiC nano-ceramic particles in stir casting followed by semisolid extrusion. Besides, it causes more spherical grains and uniform dispersion of SiC nanoparticles. Thus, Ni increases the UTS and elongation of the nanocomposites. Ti results in the formation of flake-like intermetallic phases, which negatively affect the stress concentration and ductility of the nanocomposites.

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