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THE EFFECTS OF MECHANICAL ALLOYING ON THE EXTRUSION PROCESS OF AA 6061 ALLOY REINFORCED WITH SI_3N_4

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Abstract. *The synthesis of materials by high energy ball milling of powders was first developed for the production of complex oxide dispersion-strengthened nickel alloys for structural, high temperature applications but has been attracting attention in the field of fabrication processes like the production of intermetallic compounds, supersaturated solid solutions, amorphous materials and metal matrix composites. However, due to the high level of deformation imposed, the aluminum mechanically alloyed undergo extensive grain growth during the extrusion process, resulting in serious damage in the extruded materials. This work investigates the effects of mechanical alloying on the extrusion of AA6061 aluminum alloy and the same alloy reinforced with silicon nitride. In both cases, the energy of deformed particles produced extruded bars with coarse grains in the core, while in the periphery the higher rate of deformation in the extrusion process has prevented this coarsening, resulting in a material with heterogeneous microstructure and with poor mechanical properties. This grain growth can be prevented by a higher percentage of reinforcement in the composite materials or by annealing before extrusion.*

Keywords: *Metal matrix composite, Mechanical alloying, Powder metallurgy.*

1. INTRODUCTION

The synthesis of materials by high energy ball milling of powders was first developed by John Benjamin and his coworkers (Benjamin, 1970) at the International Nickel Company – INCO. They successfully produced fine and uniform dispersions of oxide particles (Al_2O_3, Y_2O_3 and ThO_2) in nickel-base superalloys, which could not be made by other conventional powder metallurgy methods. Presently this process is being used to produce numerous materials and alloys including supersaturated solid solutions, amorphous materials, intermetallic compounds and metal matrix composites (Bose, 1995).

The process of mechanical alloying consists on repeated welding-fracturing-welding of a mixture of powder particles in a high energy ball mill. The central event is that the powder particles are trapped between the colliding balls during milling and undergo deformation and/or fracture process, depending upon the mechanical behavior of the powder components.

Mechanical alloying can be used to produce aluminum alloys and aluminum matrix composites from the elemental powders. Costa and coworkers (1996) produced AA2014 aluminum alloying from the elemental powders of cooper, silicon, magnesium, mixed in the required proportion. Lu and coworkers (1998), in the same line, produced the Al-4.5% (wt.) Cu-10% (vol.) SiC composite.

This work investigates the effects of the mechanical alloying on the extrusion of AA6061 aluminum alloy and AA6061 reinforced with silicon nitride. For this purpose, two different processes to mixture the matrix and the reinforcement powders were used: in a low energy ball mill and in a high energy attrition mill.

The process in a ball mill with low speed of rotation is classified as a low energy process, promoting only the mixture and not changing the characteristics of the powders. In a high energy attrition mill, milling occurs by the stirring action of a vertical rotating shaft with horizontal blades, which cause differential movement between the balls and the powders. Due to the high rotation speed employed, this process can be classified as a high energy process, which changes the characteristics of the powders by deforming, welding the ductile particles and fracturing the fragile ones.

However, the high deformations imposed to the mechanically alloyed powder difficult the following process of consolidation. The aim of this work is to investigate how the excess of stored energy in the material which was submitted to mechanical alloying can effect its extrusion behavior, comparatively to materials which were submitted to low energy process of mixture.

2. EXPERIMENTAL PROCEDURE

Matrix powder used as raw material was AA 6061 from The Aluminium Powder Co. Ltd, with mean size of 75 microns. Reinforcement powder used was silicon nitride from Advanced Refractory Technologies, Inc., with mean size of 8.6 microns. Three different mixture compositions were produced: 0, 5 and 15% (wt.) of silicon nitride, added to the aluminum alloy.

In the ball mill mixture process was used a horizontal ball mill with the following parameters:

- Volume: 2.5 l.
- Weight balls/powder ratio: 10/1.
- Balls diameter: 10 mm.
- Balls material: stainless steel AISI 420 quenched.
- Rotation speed: 150 rpm.
- Mixing time: 90 minutes.

In the attrition ball mill process was used a vertical attrition mill with the following parameters:

- Volume: 0.3 l.
- Weight balls/powder ratio: 6/1.
- Balls diameter: 2.5 mm.
- Balls material: stainless steel AISI 420 quenched.
- Rotation speed: 1560 rpm.
- Mixing time: 90 minutes.

Microwax (1% wt) of from Hoechst Co was added as controller agent, to prevent excessive welding of aluminum powder.

The powders were cold pressed in a cylindrical matrix at 200 MPa, with zinc stearate as lubricant. Obtained samples had 80% of the theoretical density.

The samples were then extruded with a extrusion ratio of 25/1 at 500ºC, with graphite as lubricant agent, without canning and degassing. Extruded bars with 5 millimeters diameter, 98% of the theoretical density were obtained. Half of the samples compacted with the mechanically alloyed powders were annealed at 400ºC for 4 hours, previously to the extrusion.

Therefore, extruded bars were prepared according to the conditions shown in Table 1.

Table 1 – Conditions used to produce extruded bars

Material	Mixing process	Annealing previously to extrusion
AA 6061	low energy	no
	high energy	no
		yes
AA 6061 + 5% (wt) Si_3N_4	low energy	no
	high energy	no
		yes
AA 6061 + 15% (wt) Si_3N_4	low energy	no
	high energy	no

Tensile tests to determine Ultimate Tensile Strength were performed in the extruded bars (test condition: 1.5 mm/min; room temperature). Metallographic analysis were performed in a parallel plane related to the extrusion direction, Keller´s etchant was utilized.

3. RESULTS

Figure 1 shows the microstructure of AA 6061 extruded bar. It can be observed an homogeneous structure throughout.

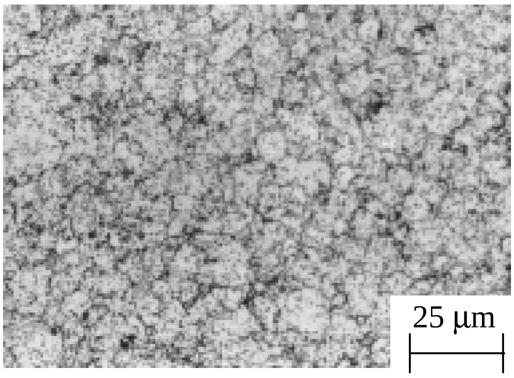

Figure 1 - Microstructure of AA 6061 extruded bar produced from powder mixed in a low energy process.

Figure 2 shows the microstructure of AA 6061 extruded bar produced from mechanically alloyed powder (high energy mixing process). It can be observed deformed grains in the peripheral zone and coarse grains in the core of the bar.

Figure 3 shows the microstructure of AA 6061 extruded bar produced from mechanically alloyed powder submitted to annealing treatment previously to the extrusion forming. Again, it can be observed peripheral zone with deformed grains and coarse grains in the center of the bar, however less coarse than the grains observed in the same region in the bar produced from material without annealing.

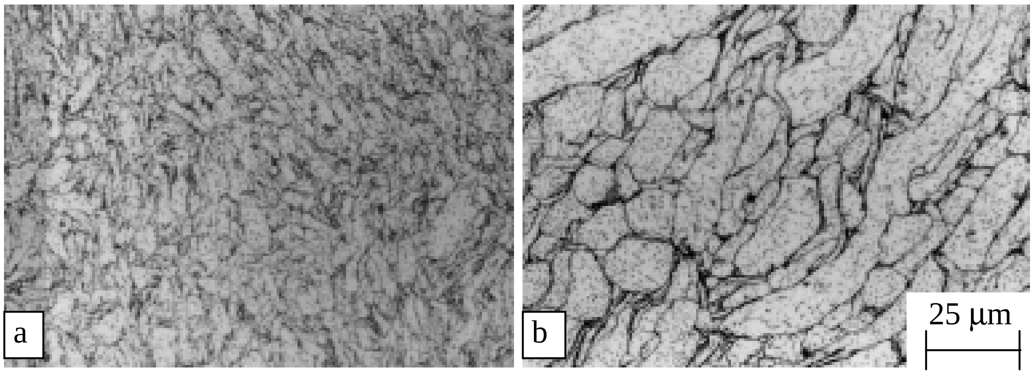

Figure 2 – Microstructures of AA 6061 extruded bar produced from mechanically alloyed powder in the (a) peripheral zone and (b) core of the bar.

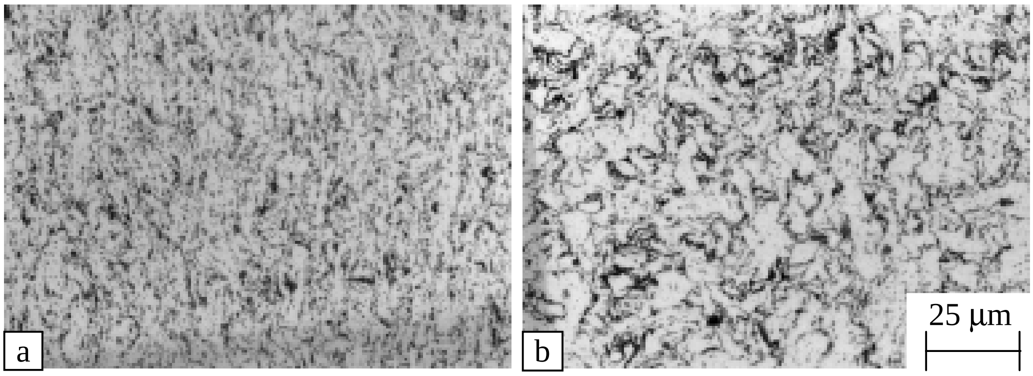

Figure 3 - Microstructures of AA 6061 extruded bar produced from mechanically alloyed powder, submitted to annealing before extrusion process in the (a) peripheral zone and (b) core of the bar.

Figure 4 shows the produced composites. It can be observed an homogeneous distribution of the reinforcement particles independent on the reinforcement percentage and the matrix/reinforcement powders mixture process.

Figure 5 shows in more detail the microstructure of extruded bar of AA 6061 reinforced with (a) 5 and (b) 15% (wt.) silicon nitride produced from powders mixed in low energy ball mill. It can be observed good distribution of silicon nitride particles and matrix grain sizes in the same order of magnitude of the grains in the bar without any reinforcing particles (see Figure 1).

Figure 6 shows in higher magnification the microstructure of AA 6061 reinforced with 5% (wt.) silicon nitride produced by extrusion of mechanically alloyed (high energy attrition mill) powders. Similarly to the extruded structure in the bars produced from mechanically alloyed powders (Figure 2), it can be observed deformed grains in the peripheral zone and coarse grains in the core of the bar. However, when compared to structures shown in Figure 2 it can be noted that the presence of the reinforcement particles seems to prevent grain growth at certain extent.

Figure 7 shows microstructures of the extruded AA 6061 reinforced with 5% (wt.) silicon nitride produced from mechanically alloyed powders submitted to annealing previously to the extrusion process. Again heterogeneous microstructure can be observed, with deformed grains in the peripheral zone and coarse grains in the center of the bar. When compared to microstructures of extruded composites produced by the same mixing process but not submitted to annealing, (Figure 6) it can be observed that annealing treatment clearly prevents grain growth in the center of the bar.

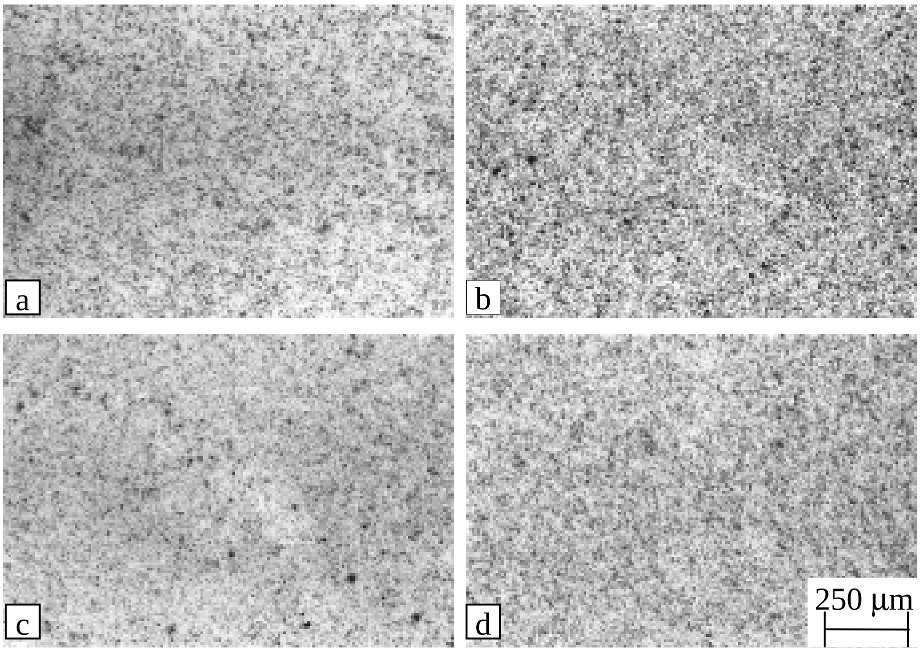

Figure 4 – Microstructures of extruded bars of AA 6061 matrix composites reinforced with (a) 5 and (b) 15% (wt.) silicon nitride produced from low energy mixed powder and of AA 6061 matrix composite reinforced with (c) 5 and (d) 15% (wt.) silicon nitride produced from mechanically alloyed powders.

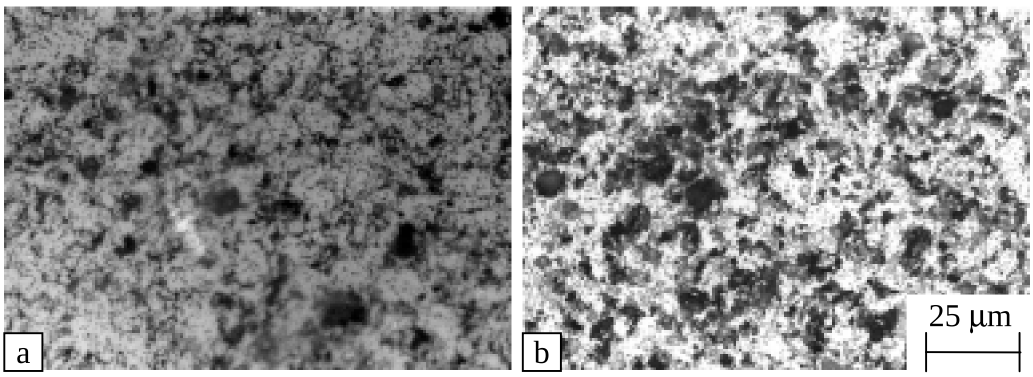

Figure 5 – Microstructures of extruded bars of AA 6061 matrix composites reinforced with (a) 5 and (b) 15% (wt.) silicon nitride produced from low energy mixed powders.

Figure 8 shows microstructures of extruded AA 6061 reinforced with 15% (wt.) silicon nitride produced from mechanically alloyed powders. When comparing to the situations presented in Figures 2 and 6, respectively extruded bars of AA 6061 and the composite containing 5% (wt.) silicon nitride produced from mechanical alloyed powders, it can be observed less coarsening of grains in the core of the bar with increasing of reinforcement content. The structure obtained is even more homogeneous than the composite containing 5% reinforcement extruded after annealing.

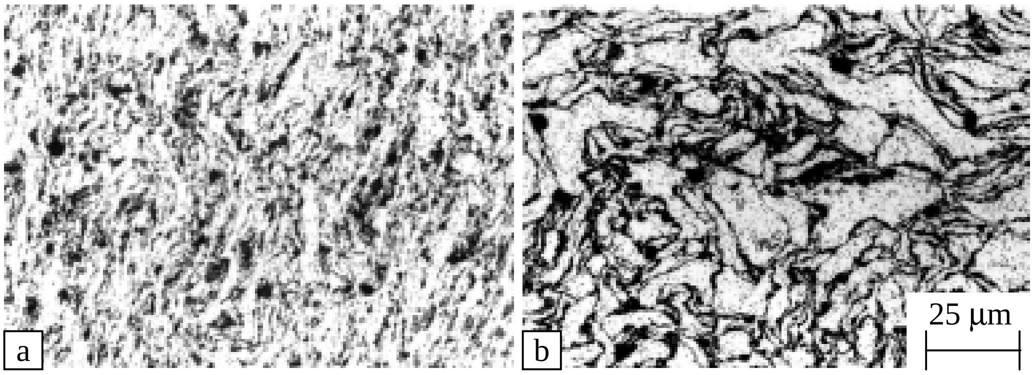

Figure 6 - Microstructures of extruded bar of AA6061 reinforced with 5% (wt.) silicon nitride produced from mechanically alloyed powders; (a) peripheral zone, (b) core of the bar.

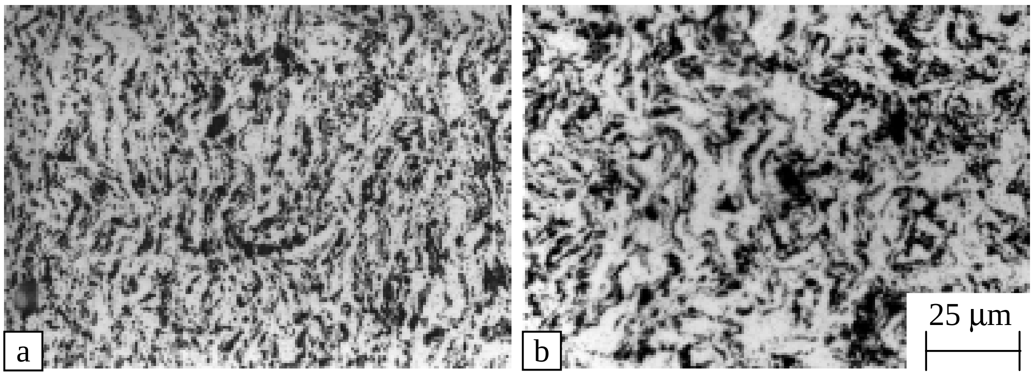

Figure 7 - Microstructures of extruded bar of AA6061 reinforced with 5% (wt.) silicon nitride produced from mechanically alloyed powders submitted to annealing previously to extrusion process; (a) peripheral zone, (b) core of the bar.

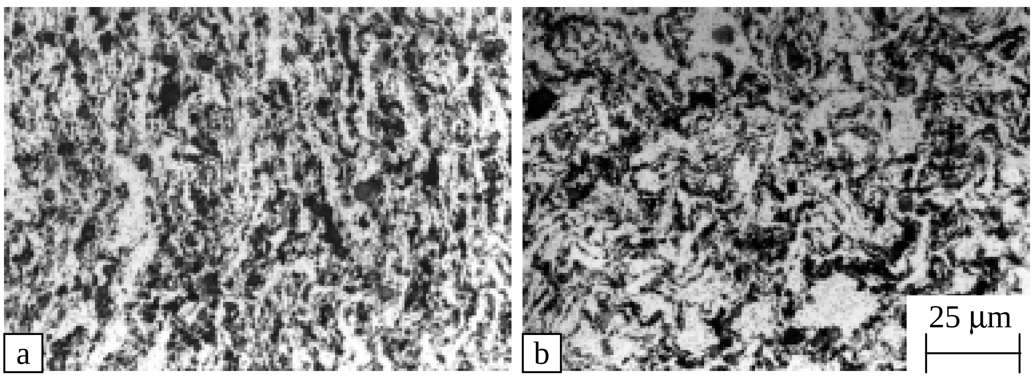

Figure 8 - Microstructures of extruded bar of AA6061 reinforced with 15% (wt.) silicon nitride produced from mechanically alloyed powders; (a) peripheral zone, (b) core of the bar.

Table 2 shows the Ultimate Tensile Strength of the extruded materials. Tensile tests were not performed in extruded AA6061 bars produced from mechanically alloyed powder due to the their unacceptable superficial condition.

Table 2 - Ultimate Tensile Strength of the extruded materials produced.

Material	UTS (MPa)
AA 6061[a]	178
AA 6061[c]	177
AA 6061 + 5% Si_3N_4[a]	200
AA 6061 + 5% Si_3N_4[b]	139
AA 6061 + 5% Si_3N_4[c]	219
AA 6061 + 15% Si_3N_4[a]	210
AA 6061 + 15% Si_3N_4[b]	181

[a] Low energy ball mill mixing process.
[b] Mechanical alloying process.
[c] Mechanical alloying plus annealing before extrusion.

4. DISCUSSIONS

A good dispersion of reinforcement particles is obtained in the extruded composites produced from powders submitted to low energy mixing as well as mechanical alloying, as observed in Figure 4. However, the high-deformed state of the mechanically alloyed aluminum highly influences the matrix's microstructure developed during the extrusion process.

The mechanically alloyed powder presents high deformed microstructure and high deformed morphology with high superficial area while the as-fabricated powder has a predominant spherical morphology. These two kinds of stored energy - the deformed structure and the high superficial area will promote the excessive grain growth during the heating of the compacted materials to the extrusion temperature.

In the extrusion process, the coarse structure so developed must be formed. The higher extrusion deformation rate in the region closer to the periphery of the bar produces a higher deformed structure in this region, resulting in final small grains, while the smaller deformation rate in the core of the extrusion neck is unable to refine the coarse grains in the same extent as in the periphery (see Figure 2 and Figure 6). Recrystallization during extrusion does not occur since aluminum and its alloys do not recrystallize dynamically at elevated temperatures; they exhibit high recovery rates and thus the dislocation density required to nucleate and drive the recrystallization does not develop (Courtney, 1990).

On the other hand, the annealed mechanically alloyed material has lower stored energy due to the recovering and recrystallization during annealing (which is performed at lower temperatures when compared to extrusion temperatures), leading to a less significant grain growth during the heating of the compacted materials to the extrusion temperature. In the extrusion process, the deformation of the recovered and recrystallized structure will produce a more homogeneous microstructure (see Figure 3 and Figure 7). The annealing treatment is able to avoid the excessive grain growth of the mechanically alloyed material during extrusion, resulting in a more homogeneous microstructure in the extruded bars. An even more homogeneous microstructure in the extruded material is produced when low energy mixing process is used, due to the predominant spherical morphology of the powder and less stored energy.

The grain growth of the extruded material obtained from mechanically alloyed powders can be avoided by the presence of the reinforcement particles which act as obstacle to the grain boundary movements. The mechanically alloyed aluminum without reinforcement shows bigger grain growth than the mechanically alloyed composite reinforced with 5%(wt.) of silicon nitride (compare Figure 2 with Figures 6 and 8). The mechanically alloyed composite reinforced with 15%(wt.) of silicon nitride shows similar structure than the extruded bars produced from mechanical alloyed and annealed material (compare Figure 8 with Figure 3). Another possible reason for reducing grain growth in the composites related to the alloy alone is that a significant part of energy in the mechanical alloying process is lost in fragmenting the reinforcing particles, reducing the available energy for matrix deformation. Bhaduri and co-workers (1996) observed a reduction in the rate of refinement with milling time of the powder Al 7010 with the presence of SiC particulate reinforcement.

The mechanical properties of the extruded materials were extremely influenced by the microstructures.

The Ultimate Tensile Strength of the extruded materials decreases when the heterogeneous microstructure is obtained. Therefore, the utilization of high energy milling of powders can interfere negatively upon the mechanical properties of extruded materials. The influence of the mixing process in the UTS is more significant as the content of reinforcing particles increases: for the alloy with no reinforcement, it was obtained fragile, unacceptable extruded bars, for the composite containing 5% (wt) Si_3N_4, the decrease in UTS was around 30%, while for the composite containing 15% (wt) Si_3N_4, the decrease in UTS was around 14% when comparing both mixing process.

As already discussed the presence of reinforcing particles leads to more homogeneous and fine structures, better mechanical properties and less dependent on mixing process utilized.

Results show that annealing can produce more homogeneous structures, with smaller grains, leading to higher values of UTS.

Related to the composites, increase in reinforcement content causes increase in UTS values related to the alloy alone. In the case of the composite reinforced with 5%(wt.) Si_3N_4, there is an increase in UTS of the extruded material produced from annealed, mechanically alloyed powders compared with the material produce from low energy ball mixed powders. This is possibly due to the refinement and better distribution of the reinforcement particles after mechanical alloying. These values are in agreement with the values obtained by Amigó and co-workers (1999) in the same system, using a low energy ball mill process of mixture in the fabrication process.

5. CONCLUSIONS

Composites AA 6061 + 5%, 15% (wt.) Si_3N_4 produced from powders mixed in low energy ball mill or mechanically alloyed present good and similar dispersions of the reinforcement particles.

However, mechanical alloying promotes excess of stored energy in the mixture, which can result in excessive grain growth in the core extruded bars, therefore heterogeneous structures are obtained, unlikely in products obtained from powders submitted to low energy mixing, which are quite homogeneous.

This grain growth can be reduced by either annealing treatments of compacted powders previously to the extrusion process or by the presence of reinforcing particles.

The UTS of the extruded materials decreases when the heterogeneous microstructure is obtained and therefore can be restored by previous annealing; the presence of reinforcing Si_3N_4 increases the UTS and tends to make this property less susceptible to the mixing process.

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