Journal
of Achievements in Materials
and Manufacturing Engineering

VOLUME 61
ISSUE 1
November
2013

Composite materials based on AlMg1SiCu aluminium alloy reinforced with halloysite particles

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Received 12.09.2013; published in revised form 01.11.2013

Manufacturing and processing

ABSTRACT

Purpose: The present work describes microstructure and technological, as well as mechanical properties of AlMg1SiCu matrix composite materials reinforced with halloysite particles by powder metallurgy techniques and hot extrusion.

Design/methodology/approach: Mechanical milling, compacting and hot extrusion successively are considering as a method for manufacturing metal composite powders with a controlled fine microstructure and enhanced mechanical properties.

Findings: A structure of newly developed composite materials reinforced with halloysite nanotubes prove that a mechanical milling process allow to improve the arrangement of reinforcing particles in the matrix material. A homogenous structure with uniformly arranged reinforcing particles can be achieved by employing reinforcement with halloysite nanotubes if short time of milling is maintained thus eliminating an issue of their agglomeration. Strong plastic deformations and fine grain size and the dispersion of halloysite reinforcing particles caused by mechanical milling is substantially reinforcing the composite materials reinforced with halloysite nanotubes as expressed with nearly a threefold increase in the hardness of composite powders as compared to the value of this quantity before milling.

Research limitations/implications: Contributes to knowledge about technology, structure and properties of aluminium alloy matrix composite material reinforced with mineral nanoparticles.

Practical implications: As the fraction of halloysite nanotubes is growing to 15%, structural changes in the powders of composite materials subjected to mechanical milling are reaching the set condition 3 times faster as compared to the matrix material.

Originality/value: It has been confirmed that halloysite nanotubes can be applied as an effective reinforcement in the aluminium matrix composites. Deformation, grain size reduction and dispersion conduce to strengthening of the composite powders.

Keywords: Powder metallurgy; Composites; Halloysite

Reference to this paper should be given in the following way:
B. Tomiczek, L.A. Dobrzański, Composite materials based on AlMg1SiCu aluminium alloy reinforced with halloysite particles, Journal of Achievements in Materials and Manufacturing Engineering 61/1 (2013) 39-46.

1. Introduction

The suitable, needed properties represent a crucial factor conditioning each time a specific application of a particular material. Advantageous mechanical properties of construction materials allow to transmit various types of loads without any damages or destructions [1]. A opinion is even presented in the paper [2] that the majority of research works carried out in the field of widely understood material engineering is concentrated on the aspects of engineering materials strengthening. In general, in metallic materials being plastic materials, strengthening consists of applying actions limiting the dislocation mobilities resulting in an increased yield point and/or strength. The numerous methods of metals and metal alloys strengthening include: metallurgic factors (grain size, composition of solid solution), hot working (precipitation hardening or hardening and tempering) and cold plastic working (deformation) [3-5]. The development of modern technologies is closely linked to an effort of developing improved engineering materials with low density while ensuring higher mechanical, operational and thermal properties and a low manufacturing cost as well as the related competitive cost of final products. It is a top priority for new material technologies and manufacturing processes to produce materials meeting the demands of manufacturers of market products at the right time and place. Efforts have been made for several decades in machinery and equipment construction, including notably modern means of transportation, especially in the aviation and automotive sector, to replace steel components with such made of light metal alloys, mainly Al [6-8] and Mg [9-11]. In order to improve the properties mentioned, technologies have been developed apart from hot working processes for introducing particles and fibres into such alloys, thus creating state-of-the-art composite materials [12]. The use of particles for reinforcement in light metals alloys, mainly aluminium, improves strength, stiffness, hardness, wear resistance while keeping relatively small density [13-15].

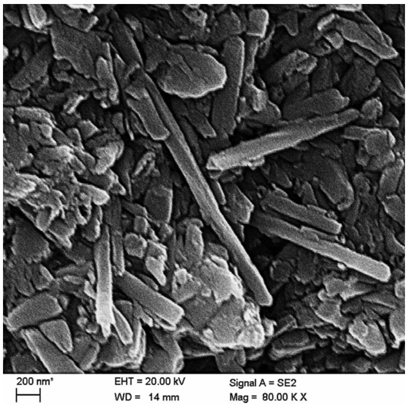

Fig. 1. Morphology of halloysite nanotubes used in the experiment

The diverse uses of composite materials with the metallic matrix results most of all from chemical and phase composition of reinforcing materials, as well as their size and fraction. The unconventional reinforcement of the composites might be the nanotubes acquired from halloysite (Fig. 1), being the clayey mineral of the volcanic origin, characteristic of high porosity, high specific surface, high ion-exchange, and simplicity of the chemical treatment and machining. Halloysite is composed of the flat surface lamellae, partially curled or in the form of tubes originating from the curled lamellae [16].

The aim of this work is to determine microstructure as well as technological and mechanical properties of aluminium AlMg1SiCu matrix composite materials reinforced with halloysite particles manufactured by powder metallurgy techniques and hot extrusion. Application of halloysite as the reinforcement of the metal composite materials is the original invention of the authors and it has been patented (PL 394587).

2. Materials and experimental procedure

2.1. Material and manufacture method

The size of reinforcing phase particles can be adjusted and the resultant mechanical properties can be improved by employing the mechanical alloying method allowing to fabricate nanostructural composite materials. Composite materials were manufactured employing as a matrix the air atomized powders of AA 6061 (AlMg1SiCu) aluminium alloy and as a reinforcement the halloysite nanotubes (Fig. 1).

A planetary ball mill (Fig. 2) was used to produce crushed and permanently joined composite powders, and three sets of specimens were prepared containing respectively 5, 10 and 15% of reinforcing particles by mass. The optimum preparation conditions for composite powders - assuming the expected structural effects - were selected based on the evaluation of morphological characteristics by means of microscopic methods, and also the tests of grain distribution of powders, their apparent density and flow rate.

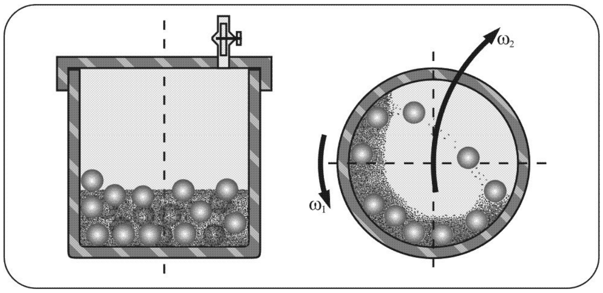

Fig. 2. Diagram of a planetary ball mill

Mechanical milling process was realised with the following parameters: charge ratio: 20:1 (wt.); ball diameter: 20mm; rotation 400 rpm, mechanical alloying time 6 hours, ball material: AISI 420 stainless steel, process control agent: 1% (wt) of microwax, no atmosphere control gas was used. To find out the outcome of the inherence of brittle reinforcement particles in the high-energy mechanical alloying process, the aluminium alloy without reinforcement was also subjected to milling.

The obtained composite powders have been compacted in the cylindrical matrix 25mm in diameter with 300MPa pressure and then extruded at 480°C with caning and without degassing.

2.2. Investigation procedure

Elaborated composite powders were characterized for their apparent density (MPIF Standard). Investigation of particles size distribution has been realized on Fritsch laser particle size analyser - Analysette 22 MicroTec Plus, based on dual laser diffraction particle sizing system. Microstructural and morphological characterization were made by scanning electron microscope SEM SUPRA 35 Zeiss. To determine microhardness suitable tests were performed with a use of the Vickers hardness tester FUTURE-TECH FM-700 under load of 50G.

3. Results and discussion

It was revealed with scanning electron microscopy that the primary globular particles (Fig. 3a) are deformed, flattened and have plate-like form in the initial stage of mechanical alloying. A propensity to connection the previous deformed particles (Fig. 4a) overcomes when milling the powders further leading to increasingly larger composite material components being formed. Changes in the shape and size of the ground powders result from individual particles being welded due to collisions with milling mediums or with the mill walls. The conglomerates of flattened particles formed as a result of welding are becoming much more reinforced, harder, and hence susceptible to cracking (Fig 5a). As milling advances, the particles are fragmented and re-joined, and this finally contributes to the random orientation of the welded particles' boundaries (Fig. 6a). A relatively equiaxial shape of the milled powder's particles informs that the process has reached the predefined status (Fig. 7a). As opposed to the AlMg1SiCu alloy powder being ground, it was found for the composite material powder that the deformed particles were tightly joined, thus creating a homogenous structure free of pores and discontinuities. Irrespective of the two times longer milling time, the AlMg1SiCu alloy powder consists of fine particles largely forming porous conglomerates with irregular, non-equiaxial shapes and the size varying within the large range of 10 to 200μm. It can be concluded by analysing a microstructure of the powders ground that a longer milling time leads to reinforcing particles being distributed homogenously, besides, powders with equiaxial particles are obtained. It also should be noted that halloysite, being a reinforcing phase of the newly developed composite materials, is undergoing brittle cracking and is strongly crushed. Investigations using the BSE detector in scanning electron microscopy allowed to confirm changes of the structure of the ground powder particles observed earlier. The cross section of AlMg1SiCu alloy powder particles in the initial condition with globular shape (Fig. 3b) is distinguished by the structure of fine, light precipitates rich in iron with the distinctive dendritic lattice-like form on the grain boundaries (Fig. 3c). In accordance with the literature [17], it is most certainly an intermetallic phase from the group $Al_{12}Fe_3Si$, Al_8Fe_2Si, $Al_{15}Fe_3Si_2$ or Al_5FeSi formed as a result of rapid cooling during sputtering.

The size of particles in the initial stage of milling is deformed, but the degree of deformation is quite differentiated. This is confirmed not only by an oval shape of some of particles but also intermetallic phase precipitates in form of a clear lattice present in its structure. The deformed particles, not considering the change of form to a plate-like shape, have their precipitates refined. The deformed, flat particles are welded many times as a result of further milling and such particles create plate structures (Fig. 4b) this way with much bigger dimensions. A specific boundary between the aluminium alloy matrix particles joined as a result of welding induced by high-energy collisions of milling mediums is represented by refined halloysite particles and also aluminium oxides coming from the surface of such particles (Fig. 4c).

The so formed large particles are becoming even twice harder as a result of strain hardening as compared to the initial state, hence very susceptible to cracking (Fig. 5b). Powder fragmentation occurring at the next stage is - apart from the hardening mentioned above - also caused by the effect of the size of large particles. Large particles are susceptible to cracking and this is related to the fact that the occurrence probability of a nucleus of cracking is directly proportional to the particle size.

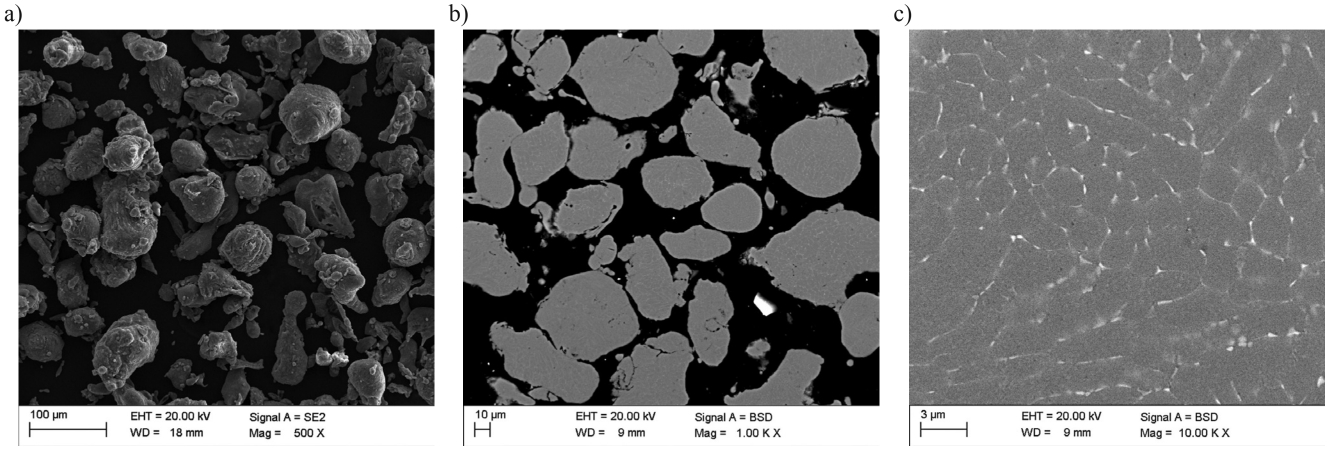

Fig. 3. Morphology (a) and microstructure (b,c) of the composite powders AlMg1SiCu + 10% of halloysite nanotubes in the initial stage, SEM

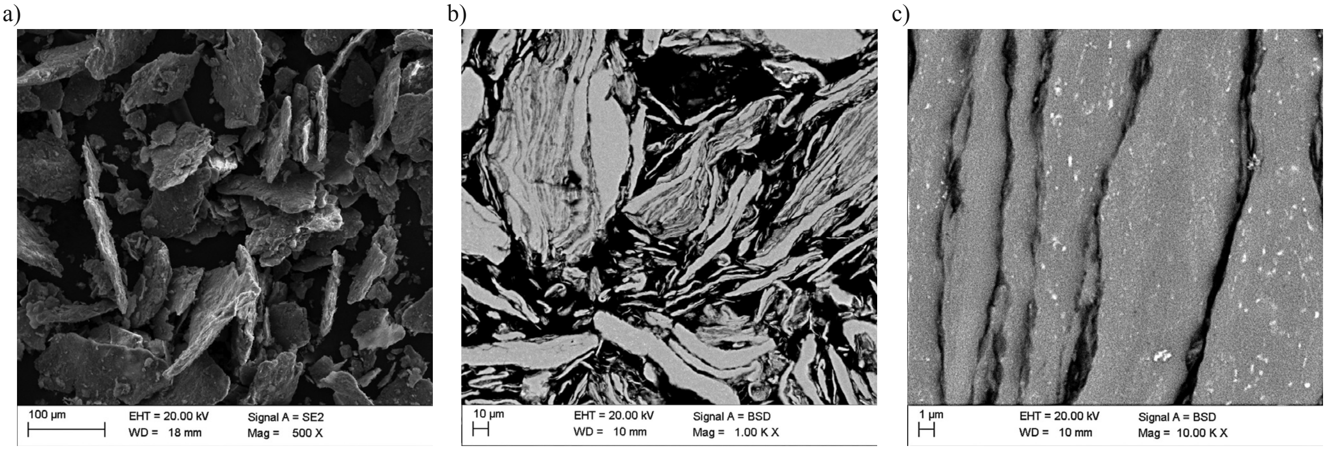

Fig. 4. Morphology (a) and microstructure (b,c) of the composite powders AlMg1SiCu + 10% of halloysite nanotubes after 90 minutes of mechanical alloying, SEM

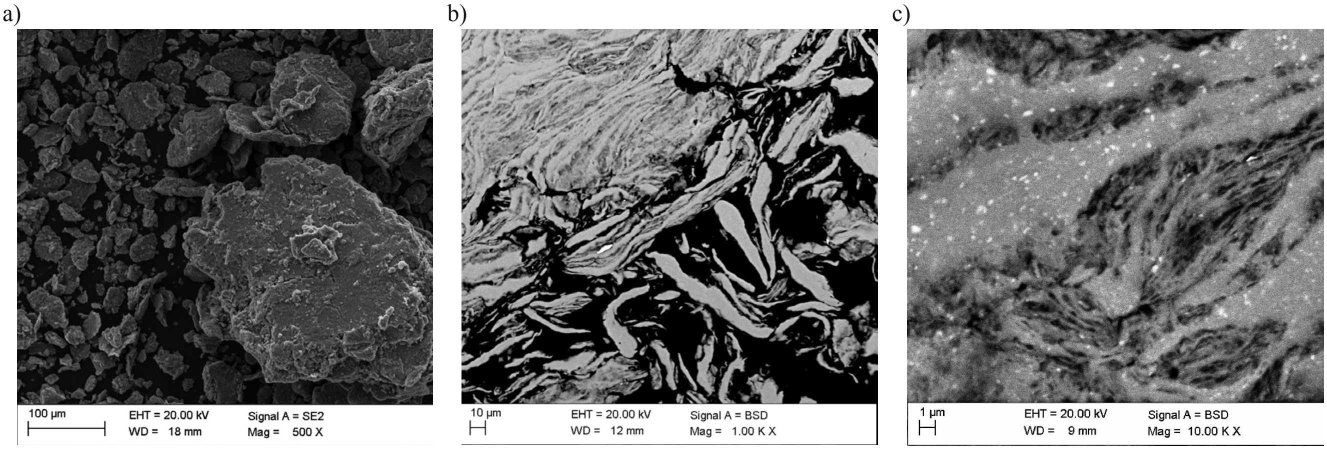

Fig. 5. Morphology (a) and microstructure (b,c) of the composite powders AlMg1SiCu + 10% of halloysite nanotubes after 180 minutes of mechanical alloying, SEM

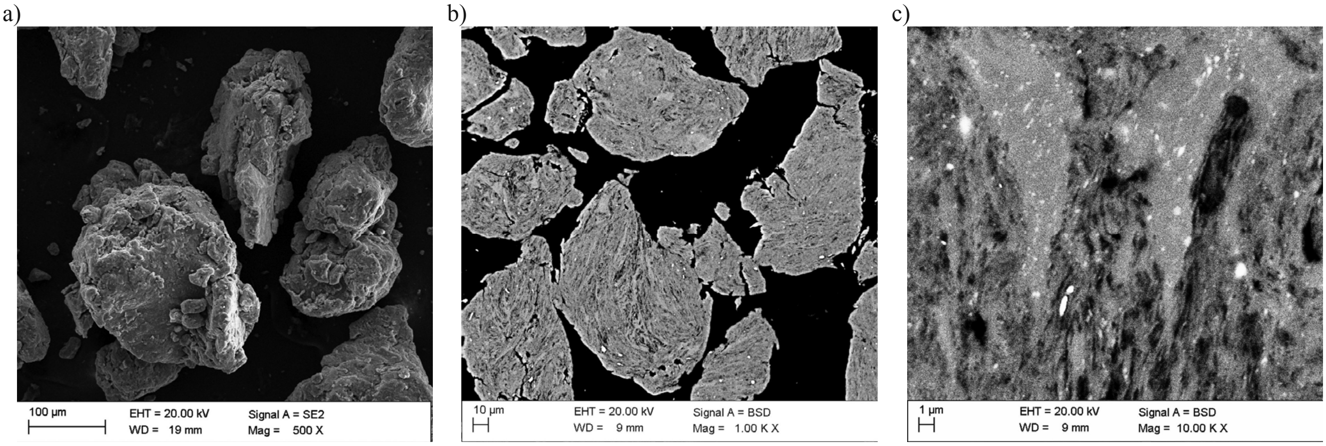

Fig. 6. Morphology (a) and microstructure (b,c) of the composite powders AlMg1SiCu + 10% of halloysite nanotubes after 270 minutes of mechanical alloying, SEM

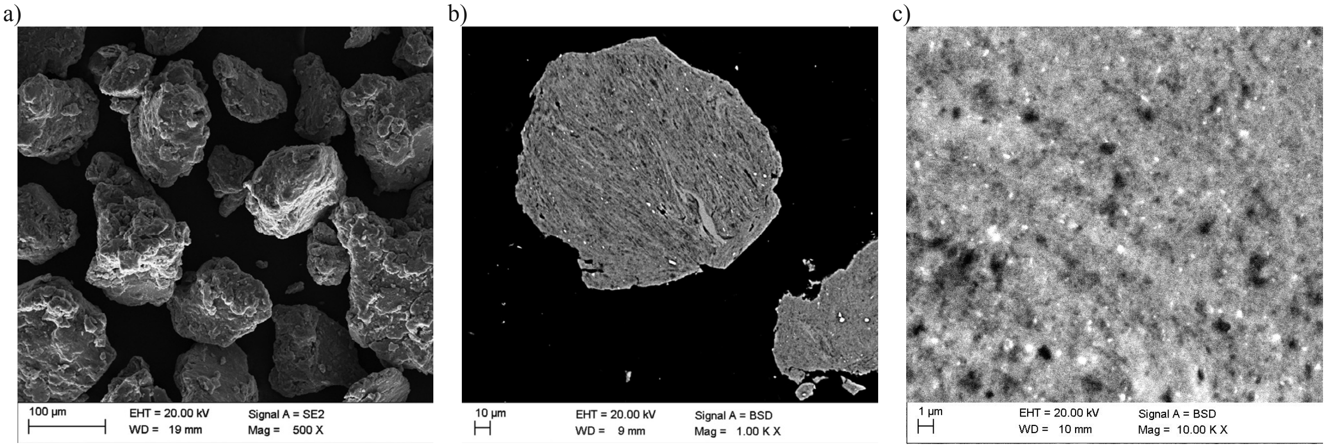

Fig. 7. Morphology (a) and microstructure (b,c) of the composite powders AlMg1SiCu + 10% of halloysite nanotubes after 360 minutes of mechanical alloying, SEM

Large, non-equiaxial plate-like particles formed as a result of overlapping and welding are - regardless their hardness - more susceptible to cracking as compared to their small sized and irregularly shaped counterparts. Fine particles are characterised by the lack of nuclei of cracks (Fig. 5c) and hence they are stronger and exhibit a tendency of inter-welding (Fig. 6b). The cracking phenomenon is accompanied by re-welding that follows such a phenomenon, and as a result - once the process is finished - this influences the random orientation of the welded particles' boundaries. A layer-like or plate-like structure from the previous stage of particular particles is consequently becoming more random due to such multiple welding and cracking, without continuous boundaries between the joined particles (Fig. 6c). The equiaxially shaped particles (Fig. 7b) and uniformly distributed fine reinforcing particles observed confirm that the process has reached the set status (Fig. 7c). Further milling does not affect largely the powder structure and properties obtained and only causes the disadvantageous sticking of particles to the mill walls and milling mediums themselves. The observed changes in morphology and structure are the same as the outcomes presented in the work [18] thus confirming the correct preparation of composite powders. It also should be noted - according to [19] - that the changes described in the structure of materials in a mechanical milling process are of the statistical nature: each of the ground particles is subject to a unique process of deformation and is characterised by an individual degree of structure refining and reinforcement.

The outcomes of microscopic observations are strengthened by an analysis of results of measurements carried out for the size of composite materials' powder particles (Fig. 8). The size distribution of the particles in the initial stage is characterised by two apexes and this is explained by the fact that the investigated powders are a mixture of particles with a largely differentiated size by also the measurement method itself. The thin, flat particles prevailing at the beginning of milling may distort the measurement results depending on the angle between the laser beam and the measured irregular particle. On the other hand, it is suggested in the work [20] that - due to a larger number of the investigated particles - ultrasound mixing of the suspension during measurement and the fact that the angle between the measured particle and the measuring beam is completely accidental, the value measured indeed corresponds to the average particle diameter. As the milling time is extending, according to the changes in morphology and structure as described earlier, a particle size distribution curve is becoming broader, thus confirming that a phenomenon exists of joining the deformed particles. The distribution curve is characterised in the next stage by an asymmetric deviation signifying a higher fraction of large particles formed as a result of multiple welding. The existing asymmetry is gradually disappearing and this is related to the cracking of large particles described earlier, and a symmetric, relatively narrow distribution curve and a median value larger by 50% than the initial state implies that the process has reached the set condition, showing the state of balance between the mechanisms of joining and fragmentation.

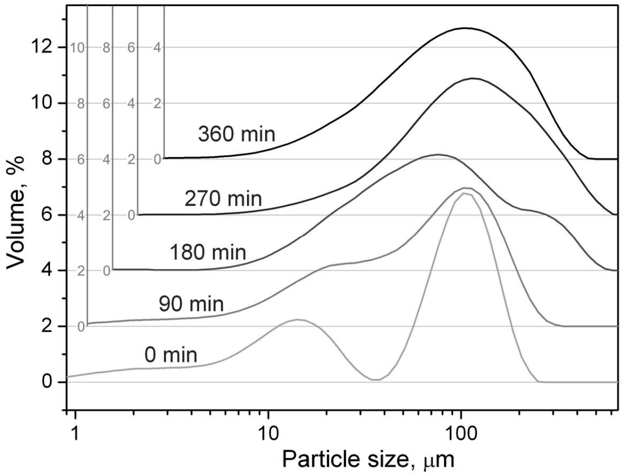

Fig. 8. Particle size distribution for the obtained composite powders reinforced with 10% of halloysite particles after different times of milling

It is found by analysing the statistical data collected in the Table 1 that the size of powder particles is subjected to constant nonlinear changes in milling.

Table 1.
Particle size measurement results of AlMg1SiCu matrix composite powders reinforced with 10% of halloysite nanotubes after different milling times

Milling time, min	Quantile $q_{0,1}$, μm	Median $q_{0,5}$, μm	Quantile $q_{0,9}$, μm
0	31.8	62.3	106.6
30	11.7	63.6	136.3
60	4.9	65.8	149.6
90	9.9	74.1	154.1
120	11.1	63.3	153.9
150	12.8	56.9	158.3
180	14.1	57.1	189.3
210	17.9	61.0	236.1
240	32.8	108.0	282.5
270	34.0	108.9	280.2
300	31.5	104.2	261.1
330	27.5	95.3	239.3
360	25.5	89.6	225.2

The apparent density of powder depends primarily on the size of particles, their shape and surface character, as well as on the degree of oxidisation and the type and quantity of gases absorbed on the surface of such particles. Considering that the reinforcing material particles possess a larger specific surface, an irregular, frequently oblong shape and form porous conglomerates, they hence have much lower apparent density as compared to globular particles, and this is consistent with [21]. Moreover, the work [22] suggest that if the reinforcing material's particles are larger than the pores between the freely filled metal matrix powder particles, they may collide with matrix powder packing, thus decreasing a apparent density value. On the other hand, fine reinforcing particles may cause the opposite effect that can be explained with a phenomenon of small particles being placed in the spaces among larger particles of a regularly shaped matrix. A diverse fraction of irregularly shaped particles is reducing maximally the overall volume as compared to, e.g. circular shapes and this results from a beneficial increase in the packing effect. The halloysite particles used are mainly characterised by much smaller particles than the AlMg1SiCu alloy matrix powder but also exhibit a strong agglomeration tendency. Having in mind that the theories mentioned are unclear and sometimes even contradictory, the key phenomenon responsible for decline in apparent density by several percents cannot be precisely pointed out by analysing the results obtained.

When two powders are plastic, as well as when one of them is brittle, it is a characteristic feature when such powders are undergoing the process that the apparent density value is changing along with the elapsing time of milling the powder of input materials mixed with each other. It was found that as the fraction of halloysite particles is growing, so is changing the dependency between apparent density and milling time (Table 2) and especially the milling time after which apparent density is rising, reaching the values higher than in the initial condition. In case of the mechanical milling of plastic aluminium powder with a brittle reinforcing phase in form of halloysite nanotubes with mass fraction of 15%, a decrease in apparent density and the following increase in apparent density occurs over the twice shorter time.

Table 2.
Apparent density of AlMg1SiCu matrix composite powders reinforced with different mass fraction of halloysite particles and after different milling times

Miling time, min	Mass fraction of halloysite particles		
	5%	10%	15%
0	1.10	1.10	1.09
60	0.63	0.72	0.88
120	0.46	0.77	1.09
180	0.58	0.96	1.16
240	0.83	1.13	1.16
300	1.11	1.14	1.16
360	1.15	1.15	1.16

The effect of brittle reinforcement fraction on the progress of the mechanical alloying process can be explained in two ways [19]. Cold plastic deformation is the driving force of the welding process occurring during mechanical milling. It should be noted though that a deformation smaller than a critical value does not cause welding, and the presence of reinforcing particles between the matrix particles during joining them is increasing the local degree of deformation, therefore, a critical value enabling welding is exceeded. In addition, local growth in the degree of deformation caused by reinforcing particles arranged in the matrix is also causing local strain hardening supporting fragmentation in the next stage. The reinforcing phases accelerating the cracking mechanisms are also expediting the process of mechanical milling. The presence of fine, hard and brittle particles acting as 'micro milling mediums' largely increasing the process energy is another phenomenon that may be decisive for an increased deformation, thus for acceleration of structural changes in mechanical milling. To summarise, the presence of reinforcing particles is supportive to a higher degree of deformation and thus accelerates milling as confirmed with apparent density investigations. Stabilisation in apparent density values shows that that the process is reaching its set condition and this correlation may also be used for determining the optimum mechanical milling process conditions. A value of apparent density after reaching the set condition, but also in further milling is achieved for a steady, slightly higher level as compared to the initial condition. Considering the statement presented in the work [21] arguing that spherical particles have the highest apparent density, then the outcomes obtained are indefinite as the sputtered aluminium alloy powder used is globularly shaped in its initial condition, and particles after the milling process are equiaxially, but irregularly shaped. Further investigations are needed to explain this aspect in detail.

The milling of powders has also decisive influence on the flow rate value of the powders developed. As demonstrated in the Table 3, powders are not flowing out of a standard funnel in the initial stage of milling due to the flaky shape of particles. Powders are considered by flow rate only at a later stage when welding processes are dominant and apparent density is rising. The results gathered in the Table 3 of flow rate tests for all the developed powders of composite materials reinforced with halloysite particles after the end of the fabrication process prove that the crucial factor prerequisite for flow rate is the method of preparation.

Table 3.
Flow rate of AlMg1SiCu matrix composite powders reinforced with different mass fraction of halloysite particles and after different milling times

Miling time, min	Mass fraction of halloysite particles		
	5%	10%	15%
0	not flow	not flow	not flow
60	not flow	not flow	not flow
120	not flow	34.21	31.98
180	35.22	24.69	23.48
240	24.9	19.23	17.81
300	17.81	16.60	15.79
360	15.79	15.99	14.98

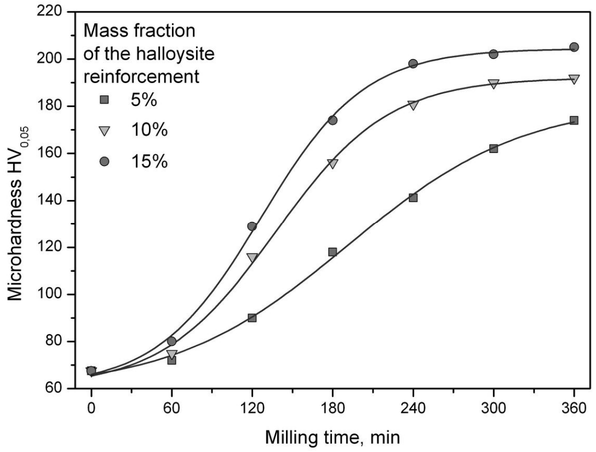

Fig. 9. Microhardness of AlMg1SiCu matrix composite powders reinforced with different mass fraction of halloysite particles and after different milling times

The use of mechanical milling leads to a high degree of deformation, which - coupled with a decreased size of grain and the dispersion of the reinforcing refined particles - reinforces the material, as best illustrated by increased hardness of composite materials powders (Fig. 9). In general, the particles of aluminium alloy powders after sputtering and those not deformed possess relatively low hardness as compared to those subjected to mechanical milling. Moreover, a standard deviation of the investigated microhardness is growing several times, especially for the milling time of 1.5÷2.5 hours as can be explained with differences in the degree of deformation between the particular particles. The reinforcement of particles in the next stage of milling is stabilised at the similar level, and after reaching the set condition - i.e. after 6 hours of milling - the hardness measured at the cross sections of particles rises nearly threefold. The results achieved confirm the information presented in the following works [18,19,23].

In conclusion, the effect of the brittle reinforcement presence in the mechanical alloying of a ductile metal, can be explained on the scheme of various stages of the mechanical alloying process in this system (Fig. 10).

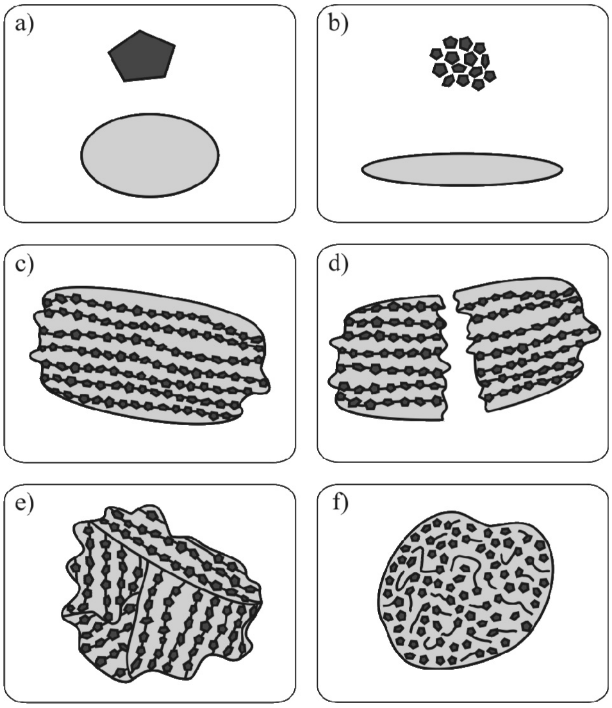

Fig. 10. The different stages of ductile-brittle system during mechanical alloying [19]

4. Conclusions

Caused by mechanical milling plastic deformations and fine grain size and the dispersion of halloysite particles is substantially reinforcing the composite materials as expressed with nearly a threefold increase in the hardness of composite powders as compared to the value of this quantity before milling. A structure of newly developed nanostructured composite materials reinforced with halloysite nanotubes prove that mechanical milling process allow to improve the arrangement of reinforcing particles in the matrix material. A homogenous structure with uniformly arranged reinforcing particles can be achieved by employing reinforcement with halloysite nanotubes if short time of milling is maintained thus eliminating an issue of their agglomeration.

Correlation was found between the fraction of a mineral reinforcing phase and the optimum conditions of manufacturing with the mechanical composite powders alloying technique: as the fraction of halloysite nanotubes is growing to 15%, structural changes in the powders of composite materials subjected to mechanical milling are reaching the set condition 3 times faster as compared to the matrix material.

Acknowledgements

The presented research was partially funded by the National Science Center as a research project No. UMO-2011/03/B/ST08/06076.

Dr Błażej Tomiczek, PhD Eng is a holder of scholarship from project POKL.04.01.01-00-003/09-00 entitled „Opening and development of engineering and PhD studies in the field of nanotechnology and materials science" (INFONANO), co-founded by the European Union from financial resources of European Social Fund and headed by Prof. L.A. Dobrzański.

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