EVALUAREA STRUCTURALĂ ŞI CARACTERIZAREA COMPOZITELOR NANOSTRUCTURATE Al-Al_2O_3 OBȚINUTE PRIN METODA ACTIVĂRII MECANICE

STRUCTURAL EVOLUTION AND CHARACTERIZATION OF NANOSTRUCTURED Al-Al_2O_3 COMPOSITE FABRICATED BY MECHANICAL ALLOYING METHOD

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This paper was focused on the structural evolution and characterization of Al-15vol.% Al_2O_3 composite. Composite powders were characterized by X-ray diffraction and scanning electron microscopy, respectively. The relationship among the stages of mechanical alloying, relative density and microstructure of both pressed and sintered materials were also investigated. It was observed that mechanical alloying process reached the steady state after 5h of milling time and relatively equiaxed powders were synthesized within this time frame. The results showed that as the milling time increased, more homogeneous dispersion of Al_2O_3 particles was obtained in the Al alloy matrix.

Lucrarea are ca scop evaluarea structurală şi caracterizarea compozitelor Al-15vol% Al_2O_3. Pulberea compozită a fost caracterizată prin difracție cu raze X şi microscopie electronică cu baleaj. Au fost de asemenea investigate relațiile dintre etapele alierii mecanice, densitatea relativă şi microstructura atât a materialului cât şi a celui sinterizat. S-a observat că procesul de activare mecanică ajunge la starea de echilibru după 5 ore de măcinare obținându-se o orientare equiaxială a pulberii în acest timp. Rezultatele demonstrează că, pe măsură ce durata de măcinare crește, se obține o mai bună dispersie a particulelor de Al_2O_3 în matricea de Al.

Keywords: *composites; mechanical alloying; sintering; porosity; hard materials*

1. Introduction

Aluminium-based metal matrix composites (Al-MMCs) are well-suited materials for structural applications in aircraft, automotive and military industries due to their high strength to weight ratio [1-3]. Al-MMCs can be produced by dispersing hard particles (such as carbides, oxides or nitrides) into the aluminium matrix either by solid or liquid techniques. Powder metallurgy (PM) is an important technique for preparation of MMCs and ceramic matrix composites (CMCs). Some of the most important advantages of PM over melting route/liquid metallurgy as follow: minimum destructive interfacial reactions between the molten metal and the second phase, no need for any kind of melting facilities to melt the matrix alloy, good distribution of the second phase and no limitation on the volume fraction of the second phase. Also, the problem of non-wet ability of second phase with molten metal does not arise [1, 4, 5]. Mechanical alloying (MA) can produce a fine and homogeneous distribution of the hardening particles in Al-based MMCs [1, 6]. Al–Al_2O_3 composites are a special class of these advanced materials because of their recent applicability in the automotive industry [7-11].

The MA is an unique process where a solid state reaction takes place between the fresh powder surfaces of the reactant materials at room temperature. Hence, it can be used to produce alloys and compounds which are difficult or impossible to be obtained by the conventional melting and casting techniques [12-15].

The aim of this study was to investigate the morphological and microstructural changes in powder particles and the influence of MA processing on the distribution of Al_2O_3 reinforcement and its corresponding properties.

2. Materials and Method

The as-received Al alloy powder with an average particle size of 377 μm (Gündoğdu Exotherm Company, Turkey) and Al_2O_3 powders (99.7 % purity, Wacker Ceramic Company, Germany) with an average particle size of 13 μm, which varies between 5 μm and 25 μm, and with a density of 3.95 g/cm^3 were used as raw materials.

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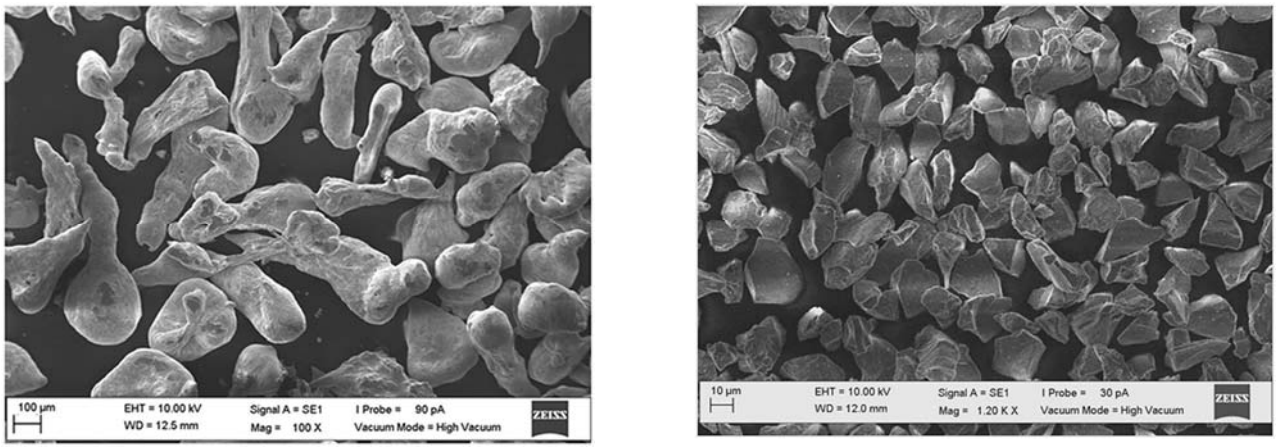

Fig. 1 - SEM images of initially used powders: (a) Al alloy, (b) Al_2O_3 particle / *Imagini SEM ale pulberilor precursoare: (a) aliaj de Al, (b) particulele de Al_2O_3.*

The chemical composition of Al alloy powder (wt. %) was as follows: Fe: 1.230, Si: 1.000, Pb: 1.000, Cu: 0.710, Zn: 0.530, Mn: 0.116, Ti: 0.071, Mg: 0.050 and Al: balance. The morphologies of both powders are shown in Figure 1(a) and (b).

The as-received Al alloy powder was ligamental while Al_2O_3 powder was polygonal in shape. The milling process was carried out at room temperature under argon atmosphere (with purity of 99.999 %) with tungsten carbide balls (diameter of 10 mm) in high-energy ball-mill (Fritsch Gmbh, model 'Pulverisette Premium line 7'). The ball to powder weight ratio and rotational speed were set to 10:1 and 400 rpm, respectively. In addition to Al alloy and powder mixture, 2 wt.% of methanol was added to the ball-mill as a process control agent (PCA).

The as-received Al alloy, the conventionally mixed (CM) and the mechanically alloyed powders were uniaxially cold pressed. Dry graphite was used in the compact process as the die lubricant in a cylindrical die at 700 MPa. The pellets were sintered at 600°C for 3h under the argon atmosphere.

Crystallite sizes of the milled powders were calculated from the X-ray diffraction line broadening using the Scherrer equation [16]. Scherrer equation can be written as follows:

$$B\cos\theta = 0.9\lambda/d \quad (1)$$

$$B = \sqrt{Bm^2 - Ba^2} \quad (2)$$

where, B is the modified peak full width, θ is the Bragg angle, λ is the wavelength of the x-radiation used, d is the crystallite size. Bm is the peak full width at maximum intensity of x-ray diffraction patterns of milled powders, and Ba is the peak full width at half maximum intensity of x-ray diffraction patterns of annealed powders.

Particle size distributions was determined by laser diffraction (Malvern, Model Mastersizer Hydro 2000e), connected to a computer that models the volume size distribution, D_{10}, D_{50}, D_{90} calculation automatically. The morphologies and distribution of the Al_2O_3 particles were analyzed by scanning electron microscopy (Zeiss Evo LS10).

3. Results and Discussion

3.1. Particle size

The effect of milling time on the particle size of ductile powders has been studied separately by previous researches in the case of monolithic and composite powders [17-22]. In all cases a similar trend was observed, an increase in the particle size followed by a decrease and then a steady state [10, 23]. However, the average particle size ($d_{0.5}$) of the monolithic Al alloy and Al alloy-15 vol.% Al_2O_3 composite powders in this study decreased continuously with the increasing milling time (Fig. 2). This can be attributed to the initially used Al alloy powders having ligamental shape. These powders can be easily deformed by high energy collisions of balls and be quickly fractured at weak regions. The particle size distribution of the monolithic Al alloy and composite powders decreased same trend with increasing milling time (Fig. 2). It found that both Al alloy and composite reached the steady state after 5h (Fig.2). The average particle size of composite powders was lower than Al alloy. The presence of alumina particles increases local deformation which improves the particle welding process. In addition, the higher local deformation imposed by reinforcement particles increases the deformation hardening, which helps the fracturing process. The small hard brittle particles in the matrix act as small milling agents, and hence milling time is reduced and steady state is obtained at much shorter time.

3.2. Morphological changes

Figure 3 shows the mechanical alloying process of two ductile metals. First, the particles undergo deformation and their morphology is changing from equiaxed to flattened. In the following stage, the welding mechanism predominates, causing the equiaxed particle formation. At this stage, oriented interfacial

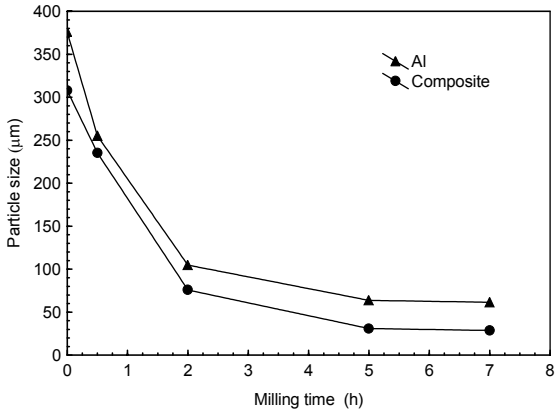

Fig. 2 - Effect of milling time on the average particle size of Al alloy and Al-15 vol.% Al_2O_3 composite powders / *Efectul timpului de măcinare asupra dimensiunii medii a particulelor de aliaj de Al şi pulberii compozite Al-15 %vol. Al_2O_3.*

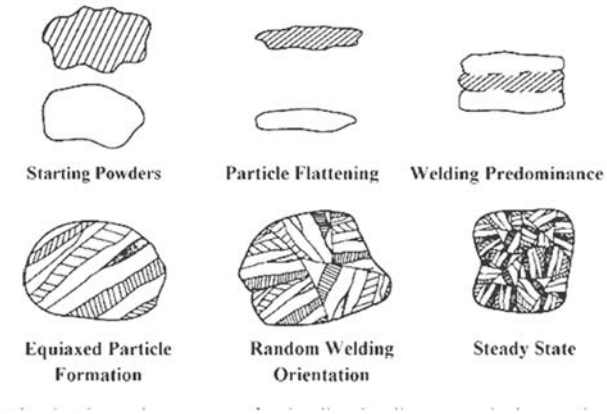

Fig. 3 - The various stages of a ductile-ductile system during mechanical alloying [19] / *Diferitele stadii ale sistemului ductil-ductil în timpul activării mecanice.*

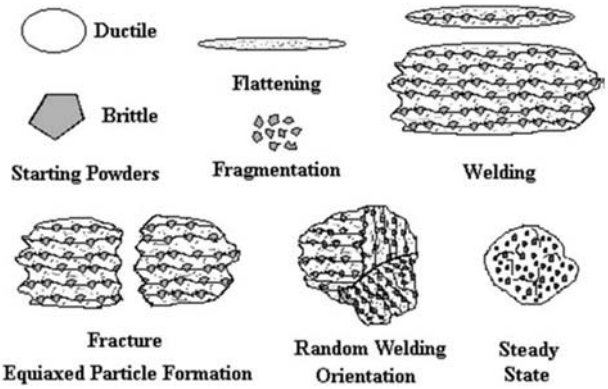

Fig. 4 - The various stages of a ductile-brittle system during mechanical alloying [19] / *Diferitele stagii ale unui sistem ductil – fragil (sfărâmicios) în timpul activării mecanice.*

boundaries are observed. Then, welding and fracture mechanisms reach equilibrium and the formation of particles with randomly oriented interfacial boundaries or, in other words, the random welding orientation occurs. At the final stage, the steady state process is observed, in which the microstructural refinement can continue, but the particle size and size distribution remain approximately the same [19].

The presence of reinforcement particles mixed with Al alloy changes the mechanical alloying process and milling classification to a ductile-brittle component system in Figure 4. We propose the following mechanical alloying process scheme for this system.

In the first stage of the milling, ductile particles undergo deformation while brittle particles undergo fragmentation. Then, when ductile particles start to weld, the brittle particles come between two or more ductile particles at the instant of the ball collision. As a result, fragmented reinforcement particles are placed in the interfacial boundaries of the welded metal particles, and the result is the formation of a real composite particle. As welding is the predominant mechanism in the process, the particles change their morphology by piling up the laminar particles. These phenomena, deformation, welding and solid dispersion, harden the material and increase the fracture process, which also contributes to the equiaxed morphology. Welding and fracture mechanisms then reach equilibrium, promoting the formation of composite particles with randomly orientated interfacial boundaries [19]. At the steady state, the microstructure undergoes a great refinement, and the interfacial boundaries are no longer visible by optical microscopy. The microstructural evolution during mechanical alloying process observed for Al alloy reinforced with Al_2O_3 is in good agreement with this model.

The variation of Al alloy-15 vol % Al_2O_3 powder shape during high energy ball milling as a function of milling time is shown in Figure 5(a–f). After the short milling of 0.5h, there is a predominance of deformed-flat powders (Fig. 5a). For composite powders the flake like Al alloy powders welded to each other and Al_2O_3 particles spaced between the layers of cold welding Al alloy powders is shown Fig. 5a. Considering the morphology changes; one can notice the effect of reinforcement particle addition on the MA process. The addition of hard reinforcement particles would accelerate the fracture process of the matrix powders, which was also reported elsewhere [23]. The increased fracturing tendency with the inclusion of reinforcement particles was due to more collision between balls and ceramic particles. After milling for 2h, the Al alloy matrix powder which was irregular and flake-like shape (Fig. 5a) became flattened and fractured (Fig. 5b) due to the impact forces exerted on the matrix powder by the grinding medium. The presence of Al_2O_3 particles in the Al alloy matrix decreases ductility; so that fracture occurs before impacts cause lamination. The Al_2O_3 particles are increasingly embedded between the Al alloy powders so preventing cold

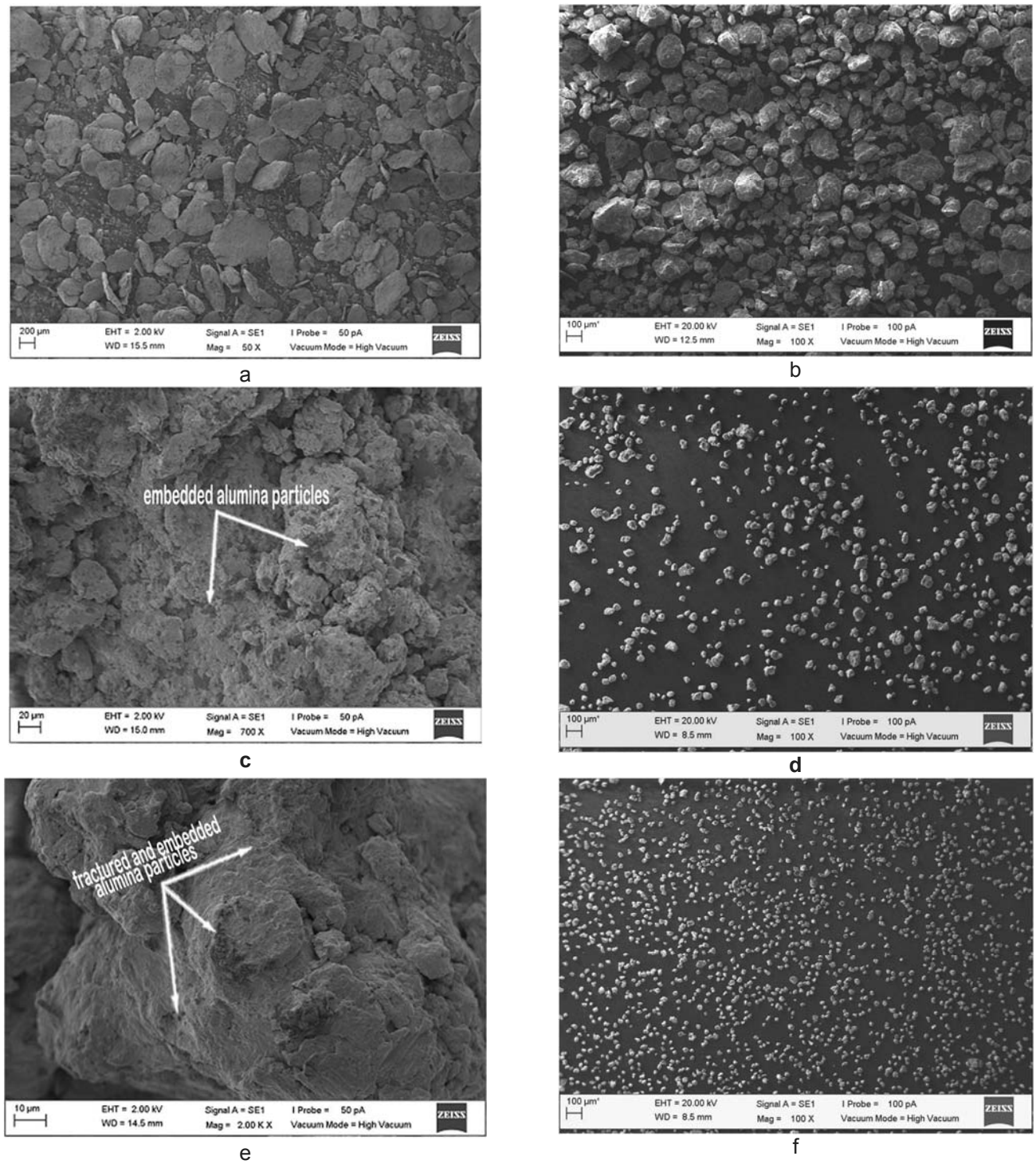

Fig. 5 - Morphology of Al alloy-15 vol.%Al_2O_3 composite powder as the function of milling time : (a) 0.5h, (b) 2h, (c) 2h, (d) 5h, relatively equiaxed powder formation (e) 5h, shows the fractured and embedded of Al_2O_3 particles on the matrix and (f) 7h, equiaxed powders (steady state) / *Morfologia pulberii compozite aliaj Al – 15 % vol. Al_2O_3 în funcție de timpul de măcinare: (a) 0.5, (b) 2h, (c) 2h, (d) 5h, formarea pulberii equiaxiale (e) 5h arată fracturarea şi încorporarea particulelor de Al_2O_3 în matrice şi (f) 7h pulberea equiaxială (starea de echilibru).*

welding from occurring with increasing milling time (Fig. 5c). Another reason is the local deformation of the Al alloy powders near of Al_2O_3 particles during the milling process. With increasing the milling time, the matrix powder shape transformed into relatively equiaxed and spherical (Fig. 5d) and the particle size became slightly reduced after 5h where cold welding domination decreased and fracturing started. Al_2O_3 particles could be dispersed inside the matrix powder after MA for a long period of time (as shown in Fig. 5e, MA for 5h). After 7h milling (Fig. 5f) the powder shape was almost equiaxed, which is the characteristic of powders at steady state.

3.3. X-Ray diffraction analysis

Figure 6 shows XRD patterns of the powders milled for various times, revealing the

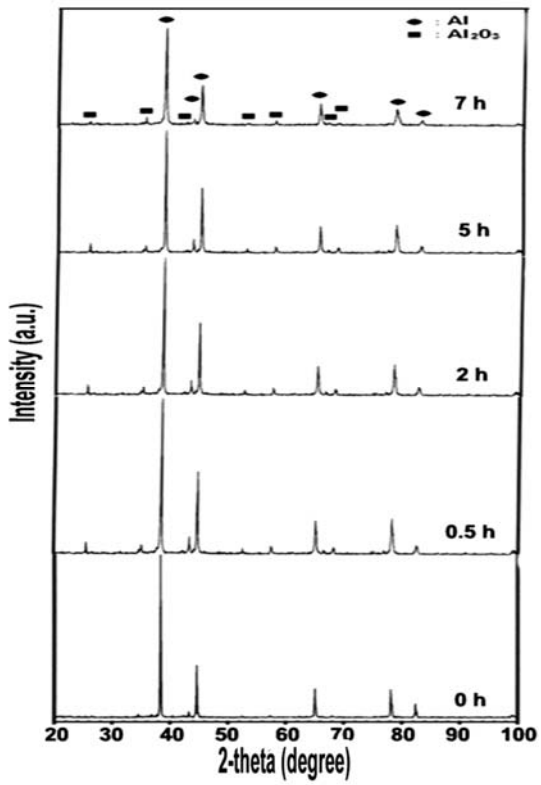

Fig. 6 - XRD patterns of the Al alloy-15 vol.% Al_2O_3 composite powder mixture for milling times: 0, 0.5, 2, 5 and 7h / *Liniile de difracție ale pulberii compozite aliaj Al – 15 % vol. Al_2O_3 în funcție de timpul de măcinare: 0, 0.5, 2, 5 şi 7h.*

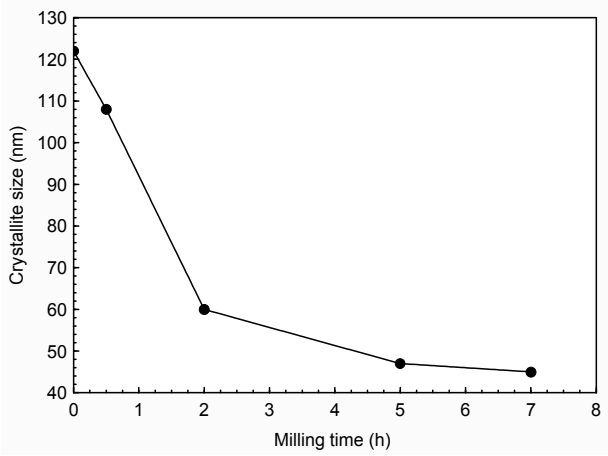

Fig. 7- Changes of crystallite size of Al-15 vol. % Al_2O_3 composite powder as a function of milling time / *Modificări ale mărimii de cristalite ale pulberii compozite Al-15% vol. Al_2O_3 în funcție de timpul de măcinare.*

structural evolution of the powder mixture as milling progressed.

By increasing the milling time, the peaks are gradually broadened and their intensities are decreased. These observations were found in agreement with the reported for other Al alloy composites [21, 23]. Analysis of the XRD patterns reveals that up to 7h milling, gradual grain refinement is the only considerable change occurring in the powder mixture and no detectable reaction takes place. A noteworthy phenomenon is the peak broadening which is due to the decrease in crystallite sizes. They were plotted versus milling time in Figure 7. The initial crystallite size of CM composite powder was about 122nm. However, the crystallite size was reduced down to 45nm after 7h milling. Figure 7 reveals that there is a plateau in the crystallite size change with milling time after 5h then crystallite reach a size of about 47nm and there after remain almost un-changed.

3.4. Structural evolution

Figure 8 shows the evolution of distribution of reinforcement particles with increasing milling time in the Al matrix. At the first stage of MA, reinforcement particles are fractured into small pieces due to their brittleness. For short milling time, reinforcement particles are not fully embedded into the Al matrix (Fig. 8a). For the second stage, as the Al_2O_3 is continuously refined, the smaller particulates start to be embedded into the matrix due to cold welding of the Al (Fig. 8b). At the final stage of MA, reinforcement particles are further refined so that they are fully embedded (Fig. 8c).

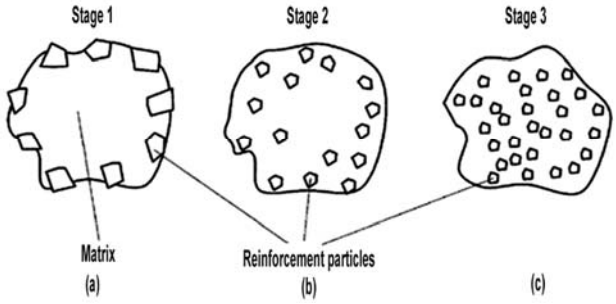

Fig. 8 - A schematic view of the evolution of distribution of the particle reinforcement reaching milling time in the Al matrix / *Model schematic al evoluției distribuției particulelor de ranforsare (umplere) în matricea de Al în timpul măcinării.*

Figure 9 shows SEM micrographs for Al_2O_3 dispersion in the Al alloy matrix after MA for different milling times. It can be seen that the complete distribution of reinforcement particles was obtained by a simple mixing method (0h milling) (Fig. 9a) which is known as the conventional powder metallurgy process. The reinforcement particles adhere together and a heterogeneous distribution of Al_2O_3 particles is obtained. Figs. 9(b-f) show that as the MA times increases, the dispersion of reinforcement particles become more homogeneous. As it was expected at the very beginning of the milling, particle distribution was not uniform and the distance between alumina particles was too much (Fig. 9b). But increasing milling time caused the big brittle alumina particles to break into smaller particles (Fig. 9c). Also, with increasing the milling time, the

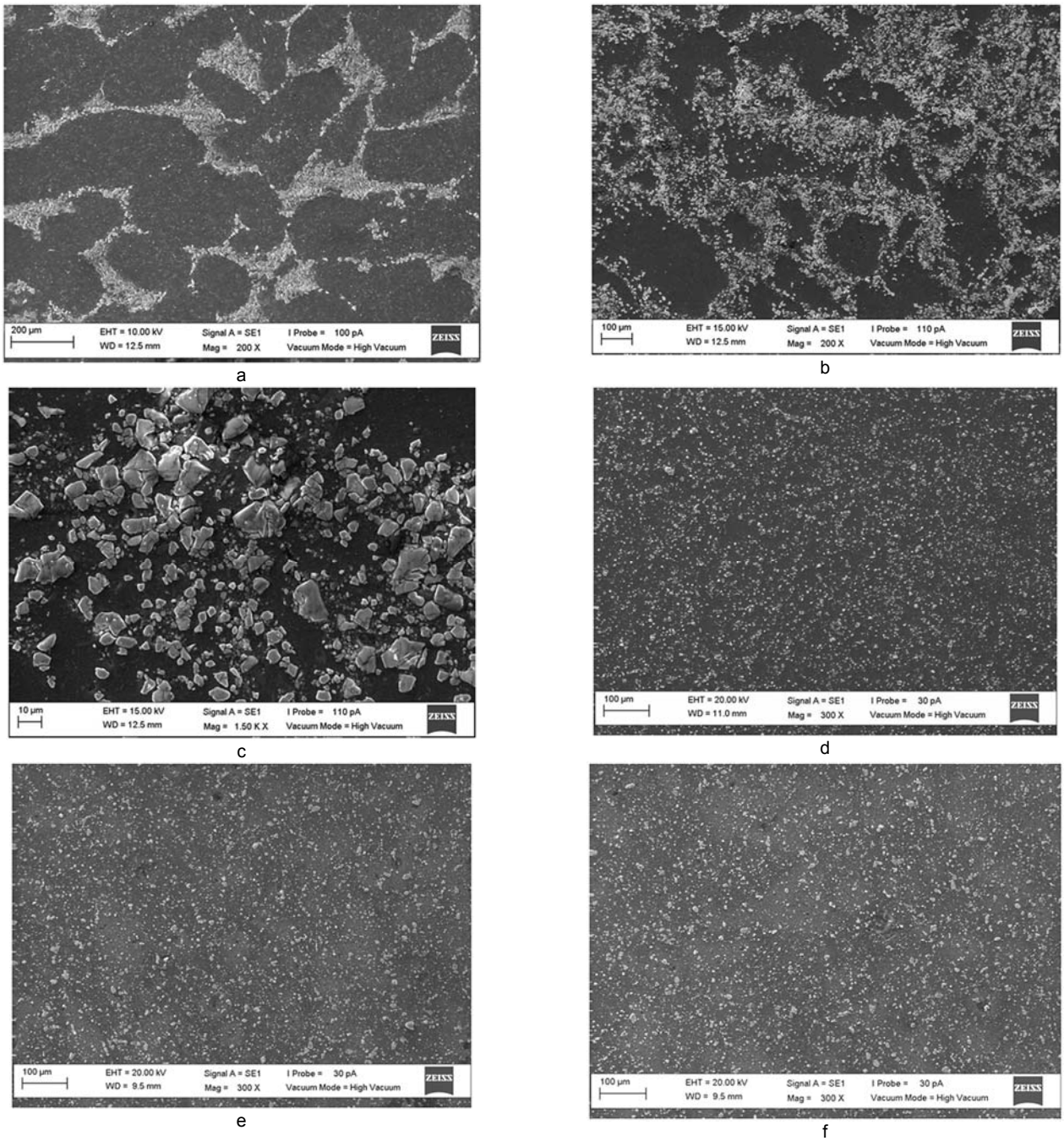

Fig. 9 - Distribution of alumina particles in the Al alloy matrix with the milling time after (a) 0h, (b) 0.5h, (c) 0.5h-high magnification, (d) (2h), (e) 5h, and (f) 7h / *Distribuția particulelor de alumină în matricea de aliaj de Al în timpul măcinării, după (a) 0h, (b) 0,5h, (c) (c)0,5 h – mărire imagine, (d) 2h, (e) 5h şi (f) 7h.*

distance between alumina particles decreased gradually (Figs. 9(d-f)). After 2h milling time (Fig. 9d), these particles were dispersed throughout the Al alloy matrix with a better homogeneity, though clustering could still be seen in some areas with precise observation of Figure 9e and 9f, one may conclude that after 5h, the milling time from 5h to 7h has not any effect on microstructure. In other words, the changes of particle size and particle distribution continue up to steady-state condition and at this condition increasing milling time has not significant effect. These results demonstrate that using MA technique to produce MMC powder produces a homogeneous reinforcement particle distribution, which cannot be achieved by using only a conventional powder metallurgy process.

Variation of relative density as a function of milling time for monolithic Al alloy and Al-15 vol.% Al_2O_3 composite are shown in Figure 10. Figure 10 shows the dependence of relative density on the milling time of the unreinforced, conventional powder metallurgy technique and mechanically alloyed composite.

It can be seen that the presence of reinforcement particles, decreased the relative density in both green and sintered samples. The relative densities for unreinforced Al alloy and composite samples decreased with increasing

milling time in the initial stage of milling, until reached a steady state value after 5h. A decrease in the densities was due to the work hardened effect of MA and hence lower deformation capacity during pressing. The green density decreases with increasing milling time due to the work hardening of the high-energy milling and the powder morphology affects the plastic deformation capacity of the powders. Spherical or equiaxed powders show lower compressibility while flattened powders show improved compressibility**.** However, according to widely accepted explanation for poor compressibility of spherical particles [26], spherical particles are subjected to compressive stress, mainly symmetrically opposed forces acting on the contact points among particles, which do not aid the sliding and cold welding. On the other hand, MA promotes work hardening and homogeneous dispersion of Al_2O_3 particles which in turn decrease the powder deformation capacity.

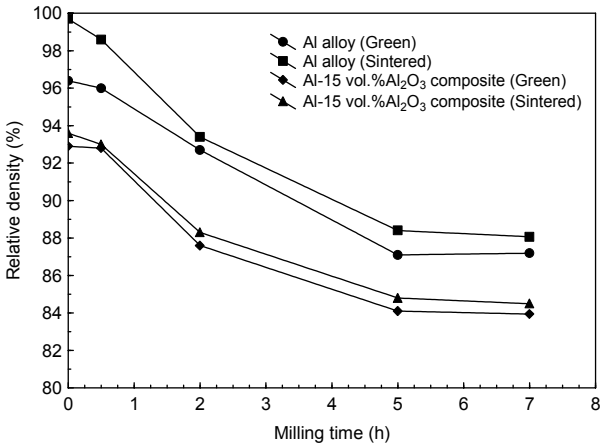

Fig. 10.- Effect of milling time on relative density of unreinforced Al alloy and Al-15 vol. % Al_2O_3 composite / *Efectul timpului de măcinare asupra densităţii relative a aliajului de Al şi compozitului Al-15% vol. Al_2O_3.*

4. Conclusions

A relatively homogeneous distribution of Al_2O_3 reinforcement in the matrix could be obtained by mechanical alloying technique at a rotational speed of 400rpm for 7h.

The Al_2O_3 particles have a great effect on the morphological characteristics of milled powder. When hard alumina particles are added to aluminium alloy powder, the fracture occurs earlier, and thus the steady-state condition is achieved after shorter milling time.

Increasing milling time leads the composite towards steady-state condition in which, all microstructure properties such as powder size, powder shape and distribution of alumina within aluminium alloy remain fixed. The distribution of the alumina powders in the Al alloy matrix reaches a full homogeneity after steady state. Besides, MA process improves the distribution and homogeneity of alumina particles in the matrix compared to conventional powder metallurgy technique process.

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NOUTĂȚI/NEWS

How smart do biomaterials need to be? A translational science and clinical point of view
Cât de moderne trebuie să fie biomaterialele? Un punct de vedere atât clinic cât şi ştiințific

Over the last 4 decades innovations in biomaterials and medical technology have had a sustainable impact on the development of biopolymers, titanium/stainless steel and ceramics utilized in medical devices and implants. This progress was primarily driven by issues of biocompatibility and demands for enhanced mechanical performance of permanent and non-permanent implants as well as medical devices and artificial organs. In the 21st century, the biomaterials community aims to develop advanced medical devices and implants, to establish techniques to meet these requirements, and to facilitate the treatment of older as well as younger patient cohorts. The major advances in the last 10 years from a cellular and molecular knowledge point of view provided the scientific foundation for the development of third-generation biomaterials. With the introduction of new concepts in molecular biology in the 2000s and specifically advances in genomics and proteomics, a differentiated understanding of biocompatibility slowly evolved. These cell biological discoveries significantly affected the way of biomaterials design and use. At the same time both clinical demands and patient expectations continued to grow. Therefore, the development of cutting-edge treatment strategies that alleviate or at least delay the need of implants could open up new vistas. This represents the main challenge for the biomaterials community in the 21st century. As a result, the present decade has seen the emergence of the fourth generation of biomaterials, the so-called smart or biomimetic materials. A key challenge in designing smart biomaterials is to capture the degree of complexity needed to mimic the extracellular matrix (ECM) of natural tissue. We are still a long way from recreating the molecular architecture of the ECM one to one and the dynamic mechanisms by which information is revealed in the ECM proteins in response to challenges within the host environment. This special issue on smart biomaterials lists a large number of excellent review articles which core is to present and discuss the basic sciences on the topic of smart biomaterials. On the other hand, the purpose of our review is to assess state of the art and future perspectives of the so called “smart biomaterials” from a translational science and specifically clinical point of view. Our aim is to filter out and discuss which biomedical advances and innovations help us to achieve the objective to translate smart biomaterials from bench to bedside. The authors predict that analyzing the field of smart biomaterials from a clinical point of view, looking back 50 years from now, it will show that this is our heritage in the 21st century.

Inovațiile din domeniul biomaterialelor şi al tehnologiei medicale din ultimele 4 decenii au avut un impact susținut asupra dezvoltării biopolimerilor, a combinațiilor oțel inoxidabil/titan şi al ceramicilor utilizate în sistemele medicale şi implanturi. Acest progres a fost generat, în primul rând, de problemele biocompatibilității şi de cerințele pentru performanțe mecanice sporite ale implanturilor permanente şi ne-permanente cât şi de sistemele medicale şi de organele artificiale. În secolul 21, comunitatea biomaterialelor are ca scop dezvoltarea unor sisteme medicale şi implanturi pentru a stabili tehnici care să satisfacă aceste cerințe şi să uşureze tratarea pacienților tineri şi vârstnici. Progresele majore din ultimii 10 ani din punct de vedere al cunoaşterii la nivel celular şi molecular furnizează fundamentul ştiințific pentru dezvoltarea celei de a treia generații de biomateriale. Prin introducerea unor noi concepte în biologia moleculară în anii 2000 şi prin progresele în genomică şi proteomică, s-a dezvoltat o înțelegere diferențiată a biocompatibilității. Aceste descoperiri biologice despre celulă au influențat semnificativ felul în care sunt concepute şi utilizate biomaterialele. În acelaşi timp au crescut cererile clinice şi speranțele pacienților. De aceea, dezvoltarea strategiilor de tratament care uşurează sau întârzie necesitatea unui implant deschid noi perspective. Aceasta reprezintă o provocare pentru comunitatea de biomateriale în secolului 21. Ca rezultat, în deceniul prezent a apărut a patra generație de biomateriale, aşa numitele materiale inteligente sau biomimetice. Un scop al conceperii biomaterialelor inteligente este înțelegerea gradului de complexitate necesar imitării matricii extracelulare (ECM) a țesutului natural. Suntem departe de re-crearea arhitecturii moleculare a ECM unu la unu şi a mecanismelor dinamice prin care informația se găseşte în proteinele ECM ca răspuns la stimulii de mediu. Printre problemele speciale legate de biomateriale se află şi un număr mare de articole al căror scop este de a prezenta starea de fapt şi perspectivele viitoare ale aşa-numitelor biomateriale inteligente, din punct de vedere ştiințific şi clinic. Scopul nostru este de a alege şi discuta care dintre progrese şi inovații ne ajută să realizăm obiectivele pentru trecerea de la ideea ştiințifică la aplicația clinică. Autorii prevăd că analiza datelor clinice de acum 50 de ani vor deveni moştenirea secolului 21.

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