VOL. 12, NO. 6, MARCH 2017 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

EFFECT ON HARDNESS AND DENSIFICATION OF MWCNT REINFORCED AA2219 COMPOSITES SINTERED IN DIFFERENT METHODS

Shijo Thomas and Umasankar. V
School of Mechanical and Building Sciences, Vellore Institute of Technology University, Tamil Nadu, India
E-Mail: shijo.thomas3@gmail.com

ABSTRACT

The current research compares the processing influence of Spark plasma sintering (SPS) over conventional sintering on aluminium alloy AA2219 - MWCNT composites. Also investigations on the effect of MWCNT content on the properties of composite are presented and discussed. Though unreinforced matrix processed under SPS showed 24.68% improvement in hardness over conventional, the same could not be realized in reinforced one due to poor bonding between the matrix and reinforcement. CNT clustering hinders proper densification during sintering and results in poor hardness; hence reinforced samples don't have a drastic change in hardness with respect to process variation.

Keywords: powder metallurgy, conventional sintering, sparks plasma sintering, metal–matrix composites, carbon nanotubes.

1. INTRODUCTION

Increased requirement of light material with enhance strength and hardness is evergreen. Carbon nano tube is focused as reinforcement on account of specific value addition to the matrix material from the point of strength, young's modulus, electrical conductivity, thermal conductivity etc. However there are challenges in getting uniform distribution and effective sintering. As automotive industry is under the pressure of improving fuel consumption and emission, specific strength, ability to work under high temperatures are search for the reinforcements to achieve these objectives are on the rise.
Aluminium alloys and its metal matrix composites (MMC) with low densities find a solution for various engineering applications [1]. Excellent fuel economy by reducing weight, Al-MMC has improved applications in transportation industries. Remarkable toughness, weldability and high aspect ratio make use of aluminum alloy AA2219 in cryogenic tanks, main body and shells for spacecraft and missiles and also in automobile component fabrication [2, 3].

Aluminium reinforced MMCs can be utilized in real life applications by overcoming the challenges in processing and achieving improved mechanical properties. Among all the processing methods for Al-CNT composites, most economic and effective technique could be the powder metallurgy (PM) route. Significant enhancement on mechanical and other properties was reported for Al-CNT composites by PM process.

High energy ball milling though reported to result in uniform mixing and distribution, it has been found that high energy ball milling of CNT and aluminium alloy results in damaging the CNT and causing agglomeration also. Hence the present study has applied sonication of MWCNT and AA2219 to achieve a uniform mixture and after drying, minimum ball milling was carried out without damaging the MWCNT.

The present work reports the synthesis of AA2219 and AA2219 with 0.75 wt% MWCNT on varying process route by conventional and spark plasma sintering (SPS). In conventional sintering heat discharge from heating element was transform to green compact by the mechanisms of conduction, convection and radiation [4,5]; where SPS [6-11] is a variation method of hot pressing with pulsed DC as a heat source and a vacuum atmospheric chamber in the presence or absence of an inert gas. Current paper discusses the findings on the effect on hardness and sintered density of varying method sintering and MWCNT reinforcement.

2. MATERIALS PREPARATION

Atomized pre alloyed aluminum alloy AA2219 powder (average particle size 42 µm) with high purity (Cu 5. 56 %, V 0.1 %, Fe 0.08 %, Mn 0.28 %, Zn 0.01 %, Zr 0.18 %, Si 0.07 %, Ti 0.07 %) from AMPAL [USA] and MWCNT (purity of >97%, aspect ratio of~1000, length of 50µm and outer diameter 20 nm) supplied by Redex [India] were used in the present study.

The distribution of CNTs in Al matrix is influenced by several factors during powder metallurgy, it includes powder size, CNT content, PCA (Process Controlling Agent) content, ball to powder weight ratio (BPR), ball milling time, milling speed (rpm), sintering type and processing technique. With higher milling intensity CNT on powder surface are embedded inside the matrix and achieving a uniform dispersion by powder plastic deformation [12]. In order to achieve uniform distribution sonication and compacting and sintering were carried out after ball milling. Received raw bundles CNT were individually dispersed in the ethanol solution by ultra sonication of about 30 minutes. AA2219 powder was added to the CNT solution and sonication was carried out subsequently for 1hour.Vaporize entire ethanol from AA2219-CNT mixture after sonication, followed by wet ball milling (using methanol as process control agent) by a laboratory planetary ball mill in 250 ml cylindrical tungsten carbide jar and tungsten carbide balls with BPR of 4:1 and at a speed of 250 rpm for 4hours. Sonication with succeed ball milling can attain a better homogeneous than ball milling alone [13].

3. EXPERIMENTAL METHODS

Premixed and ball milled powders were consolidated with different sintering techniques namely, conventional method and Spark Plasma Sintering (SPS). In conventional process milled powder mixture was compacted at room temperature in a uniaxial universal testing machine under a pressure of 450 MPa for obtain a green compact with a 25 mm diameter and 10 mm thickness, followed by pressure-less sintering in muffle furnace at 450°C for 90 minutes under nitrogen atmosphere.

The powders were placed into a die made of graphite; powder were compacted and sintered in SPS with 50Mpa and temperature of 500°c with soaking time of 5 minutes and heating rate of 100°C/min under vacuum to obtain 20 mm diameter and 10 mm thickness samples. High heating rate and limiting sintering time, SPS can limit the grain growth and Al_4C_3 formation.

4. RESULTS AND DISCUSSIONS

4.1 Density and micro hardness

Spark plasma sintered monolithic alloy and MWCNT reinforced sample have 8.9 % and 1.5% improvement in relative density respectively than a conventionally sintered sample. While adding MWCNT SPS sintering shows retardation in densification due to MWCNT agglomeration at grain boundaries, which will resist the compaction load; but conventionally sintered sample at high pressure and sintering time show improvement in densification. Optical micrographs of reinforced samples shows thick grain boundaries, indicates MWCNT agglomeration at grain boundaries. Reinforced and unreinforced sample sintered by SPS shows an improved densification and hardness than conventionally sintered samples. Figure-1 shows the density variation with respect to processing and MWCNT addition. Micro hardness (Figure-2) is in directional proportional to its density.

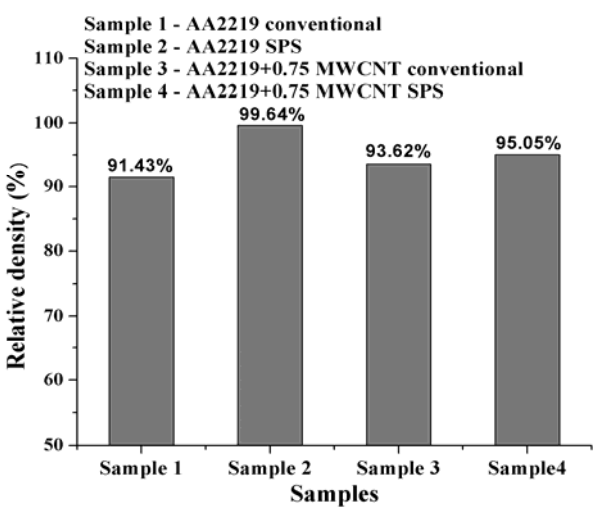

Figure-1. Relative density of conventional and SPS samples.

Increased pores content on AA2219 with MWCNT samples was due to CNT agglomeration results in resistance against compaction load and incomplete diffusion of atoms [1]. The mechanical properties were investigated with micro hardness test under ASTM standard E384−16. Micro hardness of the etched surface was measured by Vickers micro hardness tester (Shimadzu micro hardness tester, HMV - G20) with test force of HV 0.100 (980.7mN) (100gf) and a holding time of 15 seconds. Each composite indented for five times and average value was reported. Figure-2 shows the micro hardness variation in the reinforced and unreinforced samples for conventional sintering and SPS. For monolithic alloy SPS samples have marginal improvement in density. For AA2219 + 0.75wt% MWCNT both the SPS and conventional sintered samples shows almost same hardness. R. Perez-Bustamante *et al* [14] shows similar effect that hardness decrease particularly at 0.75 wt% MWCNT on varying wt% of MWCNT. In conventional addition of 0.75 wt% MWCNT the hardness was improved by about 12% and comes closer to reinforced SPS sample; where SPS reported retardation in hardness by reinforcement addition.

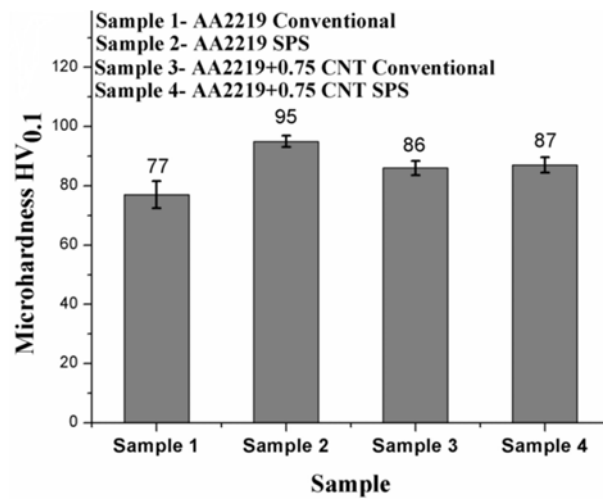

Figure-2. Micro hardness comparison of monolithic and reinforced samples processed under conventional and SPS.

4.2 Micrographs

Figure-3 shows etched optical microstructure of conventional and spark plasma sintered monolithic and reinforced samples. Comparison between conventionally and spark plasma sintered, AA2219 (Figure 3a and 3c respectively) with AA2219 + 0.75wt% MWCNT (Figures 3b and 3d respectively) shows thick grain boundaries on reinforced samples due to MWCNT accommodation. Etched optical microstructure of conventionally sintered reinforced sample (figure 3b) shows a better diffusion than spark plasma sintered composite (Figure-3d). The inappropriate bonding interface may lead to the failure of the composites. Hence the hardness of SPS samples comes down than monolithic alloy. Figure-4 shows SEM image of SPS and conventional sintered monolithic and

reinforced samples. It is to be observed that grain size in the conventionally sintered sample is comparatively more than SPS. Small grains results in improved strength and hardness as it will hinder the dislocation motions. This can be correlated with FESEM images.

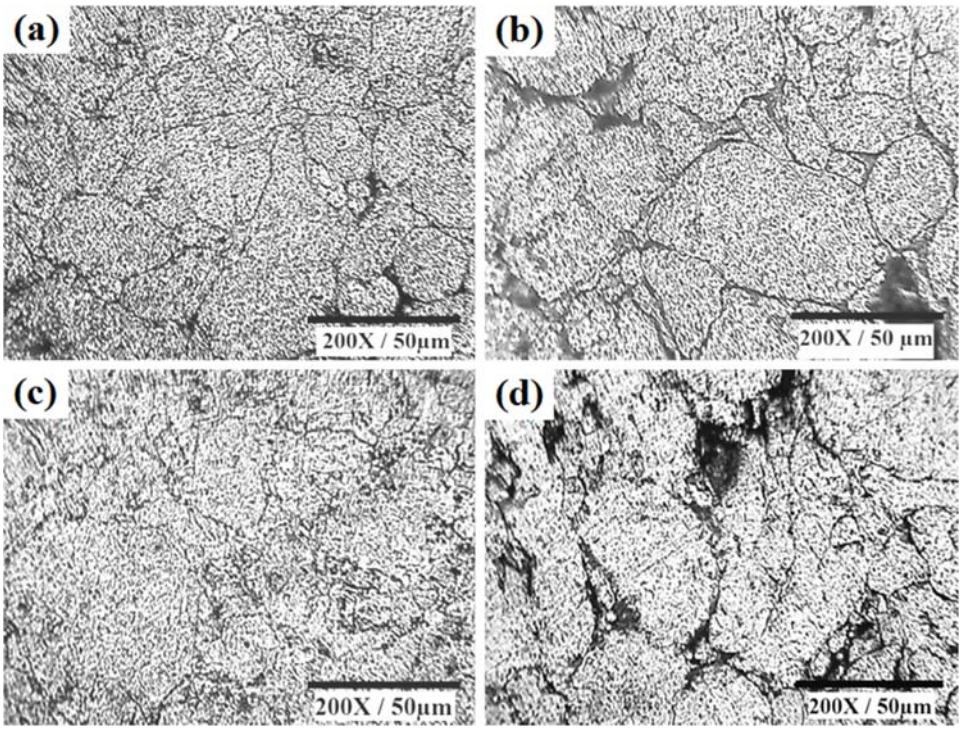

Figure-3. Etched optical microstructure of conventional sintered (a) AA2219; (b) AA2219 + 0.75 wt% MWCNT; and spark plasma sintered(c) AA2219; (d) AA2219 + 0.75 wt% MWCNT.

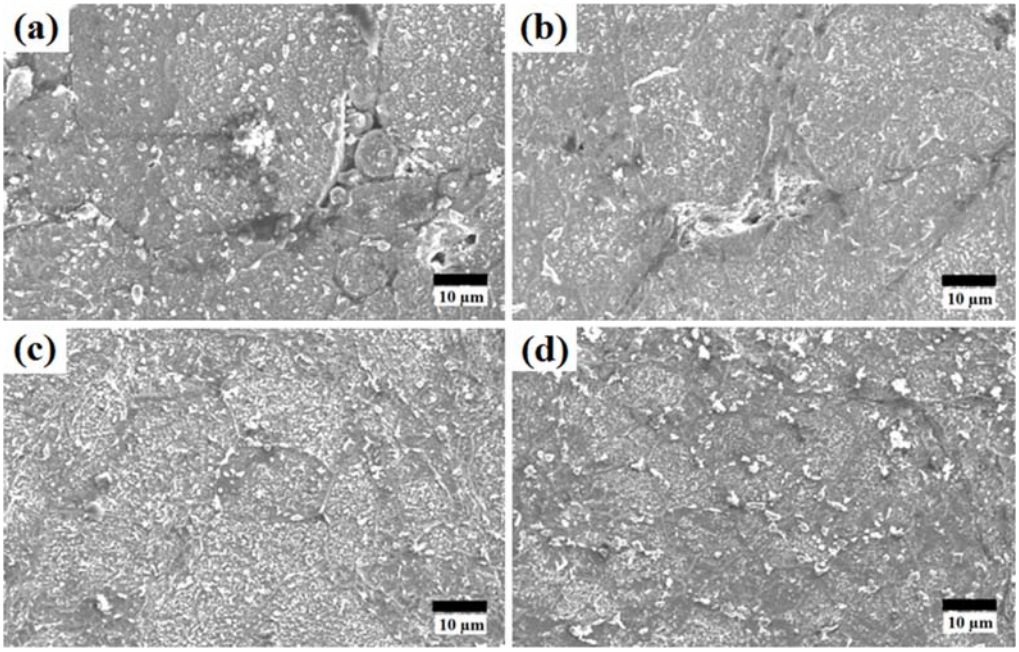

Figure-4. SEM image of conventional sintered (a) AA2219; (b) AA2219 + 0.75wt% MWCNT; and spark plasma sintered (c) AA2219; (d) AA2219 +0.75wt% MWCNT.

The microstructures of the sintered materials were investigated by FESEM (Figures 5, 6) indicating that the presence of MWCNTs after milling. Figure 5a and 6a shows homogenous distributed MWCNT in the matrix. Due to its high surface energy, MWCNT clustering cannot be avoided totally. MWCNT agglomeration (Figure 5b and 6b) will hinders proper densification results in porosity. These pores can be considered as macroscopic defects inside the material which degrade the mechanical properties of the material [1].

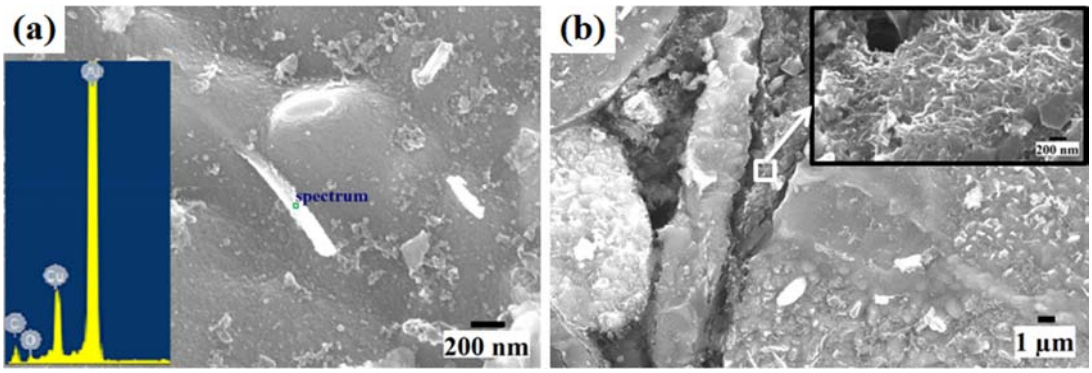

Figure 5. FESEM image of conventional sintered AA2219+0.75 wt% MWCNT showing (a) distribution of MWCNT in the matrix; (b) Clustering of MWCNT at grain boundaries.

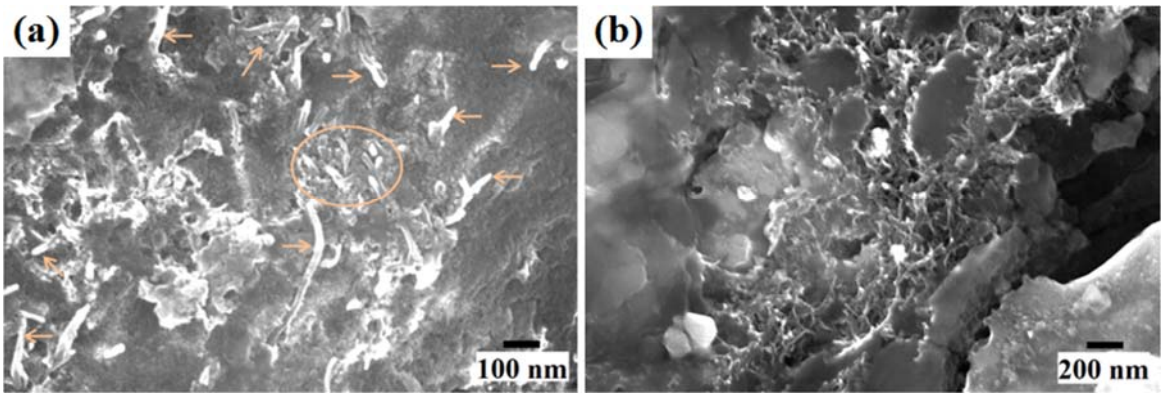

Figure 6. FESEM image of SPSed AA2219+0.75 wt% MWCNT showing (a) uniform distribution of MWCNT in the matrix (indicated by arrow mark) and agglomeration inside matrix (indicated by circle); (b) Clustering of MWCNT at grain boundaries.

4.3 XRD

Pure AA2219 alloy and alloy with MWCNT samples processed under conventional route and SPS exhibits a similar diffraction pattern. X-ray powder diffraction indicates the elemental phase conversion of Cu into Cu Al_2 and absence of Al_4C_3 during the procedure of sintering. Peak observed corresponding to Al in conventional sintered samples makes a marginal shift towards lower degree of angle after SPS.

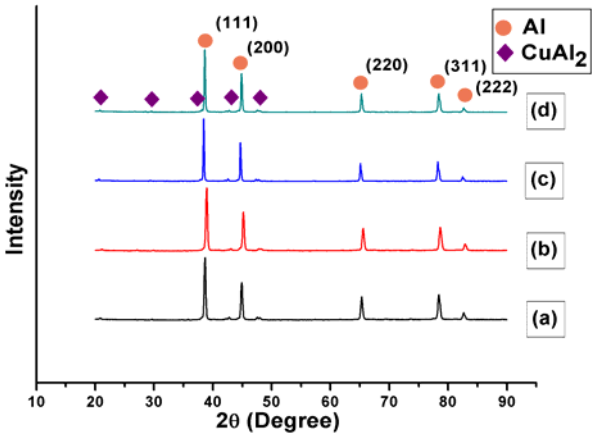

Figure-7. XRD scan of conventionally sintered (a) AA2219, (b) AA2219+0.75 wt% MWCNT and spark plasma sintered (c) AA2219, (d) AA2219+0.75wt% MWCNT.

5. CONCLUSIONS

MWCNT addition to AA2219 alloy matrix does not show a substantial improvement in mechanical properties due to lack uniform distribution. Though SPS enables increase in hardness than conventional sintering, without reinforcement due to adequate binding between the matrixes. Functional coating with enhancement of bonding with matrix material may be required to realize the advantage of matrix reinforcement. Advantage of SPS is seen with substantial increase in hardness and density. Hardness of matrix has increased to almost 24.6 % whereas the effect is not so significant with reinforcement. The reasons are clearly seen with FESEM micrographs. Efforts need to be made to achieve an effective bonding between the matrix and the reinforcement.

It was found that SPS consolidated samples showed the most promising results with improved mechanical properties, as well as the finest microstructure. Micro hardness of AA2219 is decreasing with 0.75 wt% MWCNT in SPS due to the CNT clustering and it's increasing in conventional method. 0.75 wt% MWCNT reinforced samples shows equivalent hardness in both processing route. SPS can achieve better densification and sinterability. Pores formation associated with incoherency of MWCNT-AA2219 matrix brings actual density lower than theoretical density. Low relative density and unbounded poor sinterability between matrixes is observed in conventional sintering. Absence of unbonded matrix grain in SPS pointing a good sinterability than conventional one.

REFERENCES

[1] N. Saheb, A. Khalil, A.S. Hakeem, T. Laoui, N. Al-Aqeeliand A.M. Al-Qutub. 2013. Age hardening behavior of carbon nanotube reinforced aluminum nanocomposites. Journal of Nano Research. 21. 29-35.

[2] M. Rafi Raza, Faiz Ahmad, N.Ikram, R. Ahmad and A. Salam. 2011. Development and strengthening of AA2219 alloy by mechanical working and heat treatment. Journal of Applied Sciences. 11(10): 1857-1861.

[3] P. Naga Raju, K. Srinivasa Rao, G.M. Reddy, M. Kamaraj and K. Prasad Rao. 2007. Microstructure and high temperature stability of age hardenable AA2219 aluminium alloy modified by Sc, Mg and Zr additions. Materials Science and Engineering A. 464: 192-201.

[4] M. Shukla, Sumana Ghosh, Nandadulal Dandapat, Ashis K. Mandal and Vamsi K. Balla. 2016. Comparative study on conventional sintering with microwave sintering and vacuum sintering of Y2O3-Al_2O_3-ZrO_2 ceramics. Journal of Materials Science and Chemical Engineering. 4: 71-78.

[5] M. Oghbaei and O. Mirzaee. 2010. Micro wave versus conventional sintering: A review of fundamentals, advantages and applications. Journal of Alloys and Compounds. 494: 175-189

[6] Nouari Saheb. 2014. Sintering behavior of CNT reinforced Al6061 and Al2124 Nanocomposites. Advances in Materials Science and Engineering. 2014: 1-9.

[7] Abdullah Khalil1, Abbas Saeed Hakeem and Nouari Saheb. 2011. Optimization of process parameters in spark plasma sintering Al6061 and Al2124 aluminum alloys. Advanced Materials Research. 328-330: 1517-1522.

[8] NouariSaheb, Zafar Iqbal, Abdullah Khalil, Abbas Saeed Hakeem, Nasser Al Aqeeli, TaharLaoui, Amro Al-Qutub and Ren´e Kirchner. 2012. Spark plasma sintering of metals and metal matrix nanocomposites: a review.Journal of Nanomaterials. 2012: 1-13.

[9] Zhen-Feng Liu, Zhao-Hui Zhang, Ji-Fang Lu, Alexander V. Korznikov, Elena Korznikova and Fu-Chi Wang. 2014. Effect of sintering temperature on microstructures and mechanical properties of spark plasma sintered nanocrystalline aluminum', Materials and Design. 64: 625-630.

[10] G.A. Sweet, M.Brochu, R.L. Hexemer Jr, I.W. Donaldson and D.P. Bishop. 2014. Microstructure and mechanical properties of air atomized aluminum powder consolidated via spark plasma sintering. Materials Science & Engineering A. 608. 273–282.

[11] S. Rudinsky, J.M. Aguirre, G.Sweet, J. Milligan, D.P. Bishop, M. Brochu. 2015. Spark plasma sintering of an Al-based powder blend. Materials Science & Engineering A. 621: 18-27.

[12] H.J. Choi, J.H. Shin and D.H. Bae. 2012. The effect of milling conditions on microstructures and mechanical properties of Al/MWCNT composites. Composites: Part A. 43: 1061-1072.

[13] A.M.K. Esawi, K. Morsi, A. Sayed, M. Taher and S. Lanka. 2010. Effect of carbon nanotube (CNT) content on the mechanical properties of CNT-reinforced aluminium composites. Composites Science and Technology. 70: 2237-2241.

[14] R. Perez-Bustamante, C.D. Gomez-Esparza, I. Estrada-Guel, M. Miki-Yoshida, L. Licea-Jiménez, S.A. Pérez-Garcia and R. Martínez-Sanchez. 2009. Microstructural and mechanical characterization of Al–MWCNT composites produced by mechanical milling. Materials Science and Engineering A. 502: 159-163.