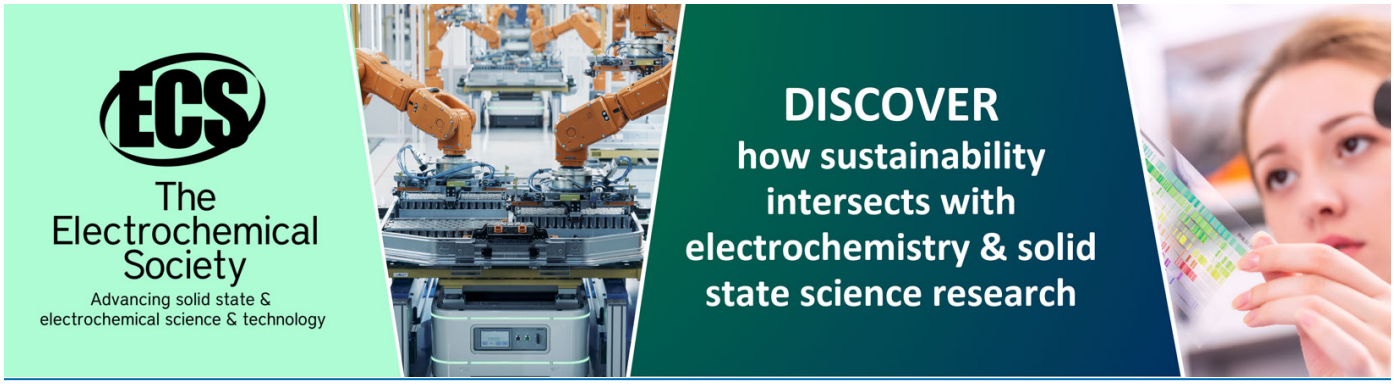
Characterization of Ball Milled Al-Al_2O_3 sub-micron Composites



Characterization of Ball Milled Al-Al_2O_3 sub-micron Composites

E. Dagasan [(a)]***, E. Gercekcioglu*** [(b)]***, S. Unalan*** [(b)]
[a] *Erciyes University, Engineering Faculty, Department of Materials Science and Engineering, 38039 Kayseri, Turkey*
[b] *Erciyes University, Engineering Faculty, Department of Mechanical Engineering, 38039 Kayseri, Turkey*

Abstract

The purpose of this study is to investigate properties of the composite powders produced by ball milling process. Different weight ratio of high purity Al_2O_3 powders were added to the Al matrix as reinforcing element. Ball milling process was conducted by a planetary type ball mill with WC milling balls and vial at constant parameters like rotating speed, time, Ball-to-Powder ratio and Process Control Agent. Samples that taken from the powder mixtureby various time intervals were analyzed by SEM, XRD and BET surface area and porosity measurement systems.

Keywords: Mechanical Milling, Aluminium Matrix Ceramic Reinforced Composites, Al_2O_3, Ball Milling.

1. Introduction

Mechanical alloying (MA) allows production of homogeneous materials starting from blended elemental powder mixtures [1, 2]. The process is generally achieved by the association of high-energy ball mills.

Mechanical Alloying (MA) is a process that consists of repeated cold welding and fracturing mechanisms of powders (Figure 1.b). And mainly used for creating alloys without melting. Additionally, this technique enable economical and rapid preparation of nanocomposites and ceramics [3]. Although there are different methods to fabricate composite materials, powder metallurgy enables net-shape and cost-effective processing with controlled microstructure and mechanical properties [4].

By the way particulate-reinforced aluminum MMCs have found an extensive commercial application in several sectors namely aerospace, space, automotive and structural [5]. This shows an excellent combination of mechanical and physical properties such as the specific strength and the stiffness, the wear resistance, and the electrical and the thermal conductivities [6, 7, 8].

Many methods are suitable for preparing aluminum matrix composites. In the liquid methods particles are added to the liquid aluminum by stirring before casting [9]. However, the difference in thermal expansion coefficients between ceramic particles and molten Al constituents and the poor wettability between these two become an obstacle to the liquid method for synthesizing Al matrix composite [10]. That makes MA advantageous for the production of Al-MMCs. Many ceramic particles like Al_2O_3

[11], SiC [12], B_4C [13, 14] and SiO_2 [15] are used as reinforcement for Al and its alloys for making properties better.

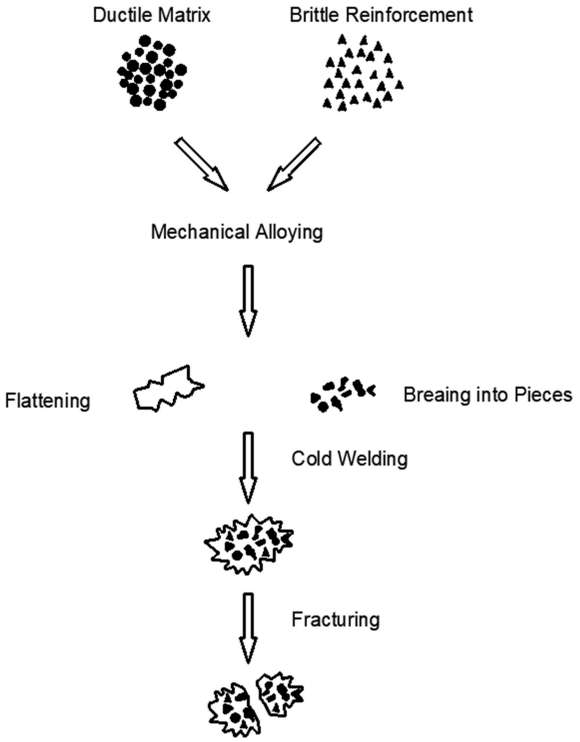


Figure 1. Schematical View of Mechanical Alloying Process.

2. Experimental Details

2.1. Milling Operations

High purity Al matrix was reinforced with high purity Al_2O_3 powders by using a planetary ball mill. Properties of the initial powders are given in Table 1.

Table 1. Properties of the Initial Powders.

	Purity	**Particle Size**
Al (Alfa Aesar- Al 00010)	99,8 %	-40 +325 mesh
Al_2O_3 (AEE-AL 602)	99,9 %	- 325 mesh

To investigate the effect of Al_2O_3 on the properties of composite powder 5, 10, 15 and 20 wt. % were added to the Al matrix. Ball milling process was conducted by the help of a FRITSCH Pulverisette 6 type planetary ball mill by using tungsten carbide vial and balls. Al-Al_2O_3 mixtures were mixed and loaded into the vial under high purity argon atmosphere by using a MBRAUN GB-2202-PVAC glove box. Parameters that are kept constant for all specimens are given in Table 2.

Table 2. Constant Milling Parameters.

Rotational Speed	250 rpm
Total Ball Weight	336 gr
BPR (Ball-to-Powder Ratio)	10:1
Total Powder Weight	33,6 gr
Vial Capacity	250 ml WC
Milling Atmosphere	High Purity Argon
PCA (Process Control Agent)	% 1.5 Stearic Acid
Total Milling Time	120 hrs

Milling operations were stopped by time intervals of 30 hours to take samples from the powder mixture. Taken samples were then used for SEM, X-RD and BET surface area analyzes.

2.2. SEM and Morphological Studies

To evaluate the morphological changes during ball milling SEM micrograms of specimens were taken by a LEO 440 type scanning electron microscope. Particle size analyzes were then realized on the SEM images by the help of an image processing software named Image ProPlus.

2.3. X-RD Analyses

X-RD analyses were conducted by a Bruker AXS D8 Advance type analyzer with Cu Kα radiation (λ=0,15406 nm). XRD patterns were recorded in the 2θ range 10-90°.

2.4. BET Surface Area and Pore Size Analyses

Surface area and pore size analyses of the composites were measured by a Micromeritics Gemini VII type analyzer. Both single point and multi point results were conducted by using nitrogen as adsorbent.

3. Results

3.1. SEM and Morphological Studies

SEM micrograms of as-received Al and Al_2O_3 are given in Figure 2.

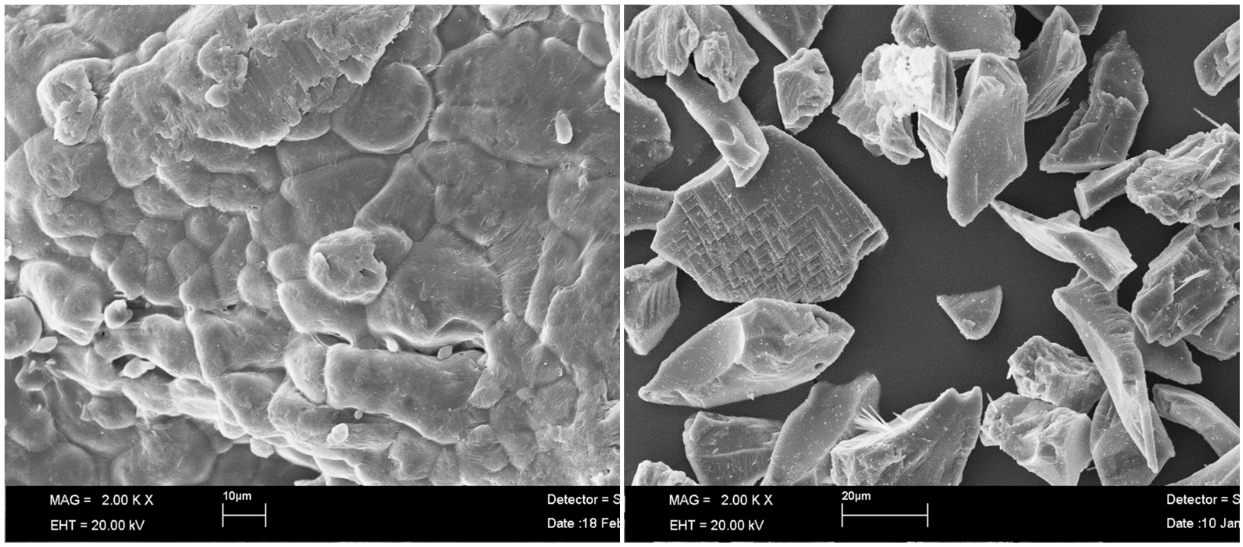


Figure 2. SEM Images of As-Received (a) Al, (b) Al_2O_3.

Images of Al-% 5 Al_2O_3 powder mixture that is ball milled for 30, 60, 90 and 120 hours are given in Figure 3.

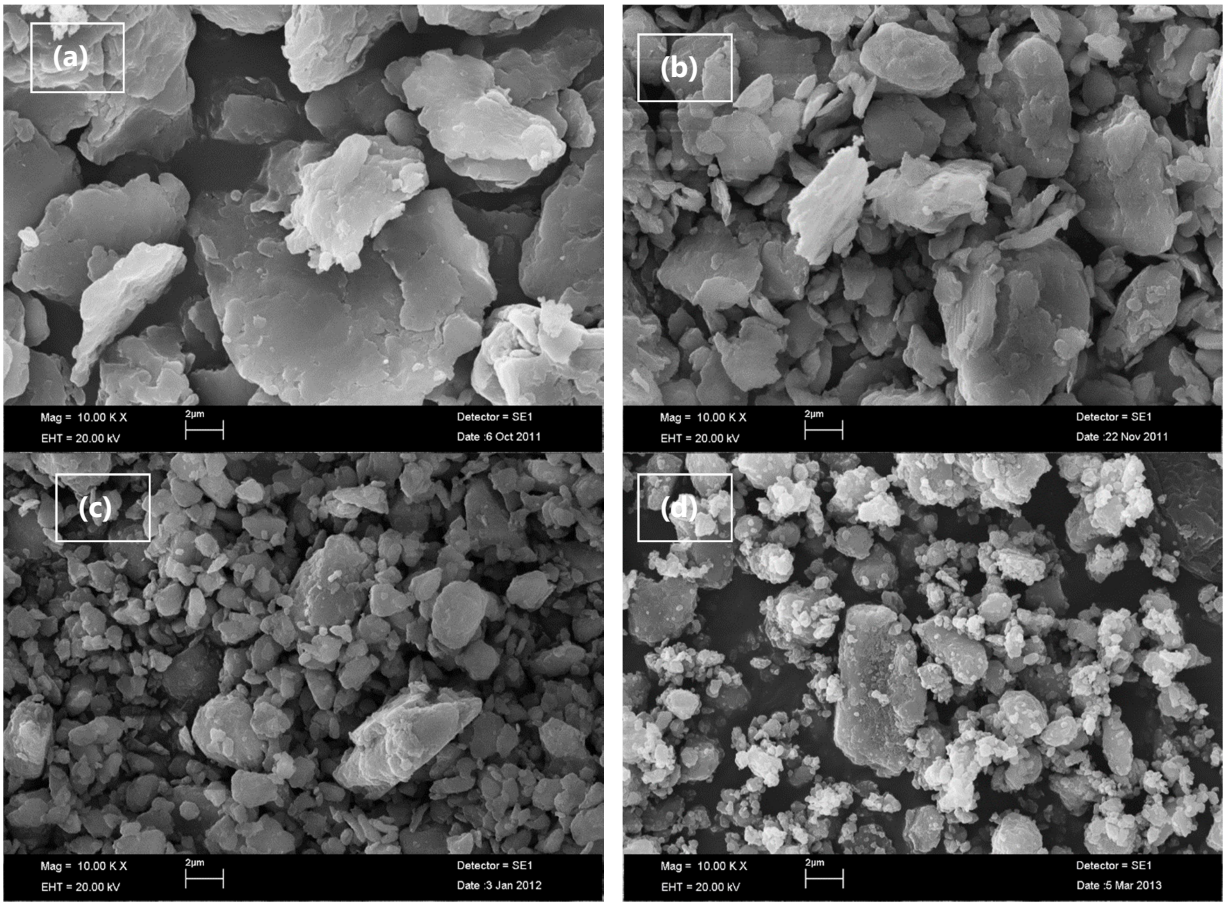


Figure 3. SEM Images of the Al- 5 % Al_2O_3 Powder Mixtures Milled for (a) 30 Hours, (b) 60 Hours, (c) 90 Hours and (d) 120 Hours.

It is clear that at initial stages of the milling process (Figure 3.a and 3.b), the powder mixture tend to flatten because of the ductile Al matrix and also the number of the broken particles of brittle reinforcement is less. At the same time it can be also said that particles are sharp cornered at initial stages. But as the milling time goes further particles became round and homogenized by means of particle size.

Figure 4, shows the morphological evaluation of Al-% 10 Al_2O_3 composite powder. As the previous sample flattening tendency goes on. But due to the increase in the weight ratio of Al_2O_3 an increase in the number of broken Al_2O_3 particles is also observed.

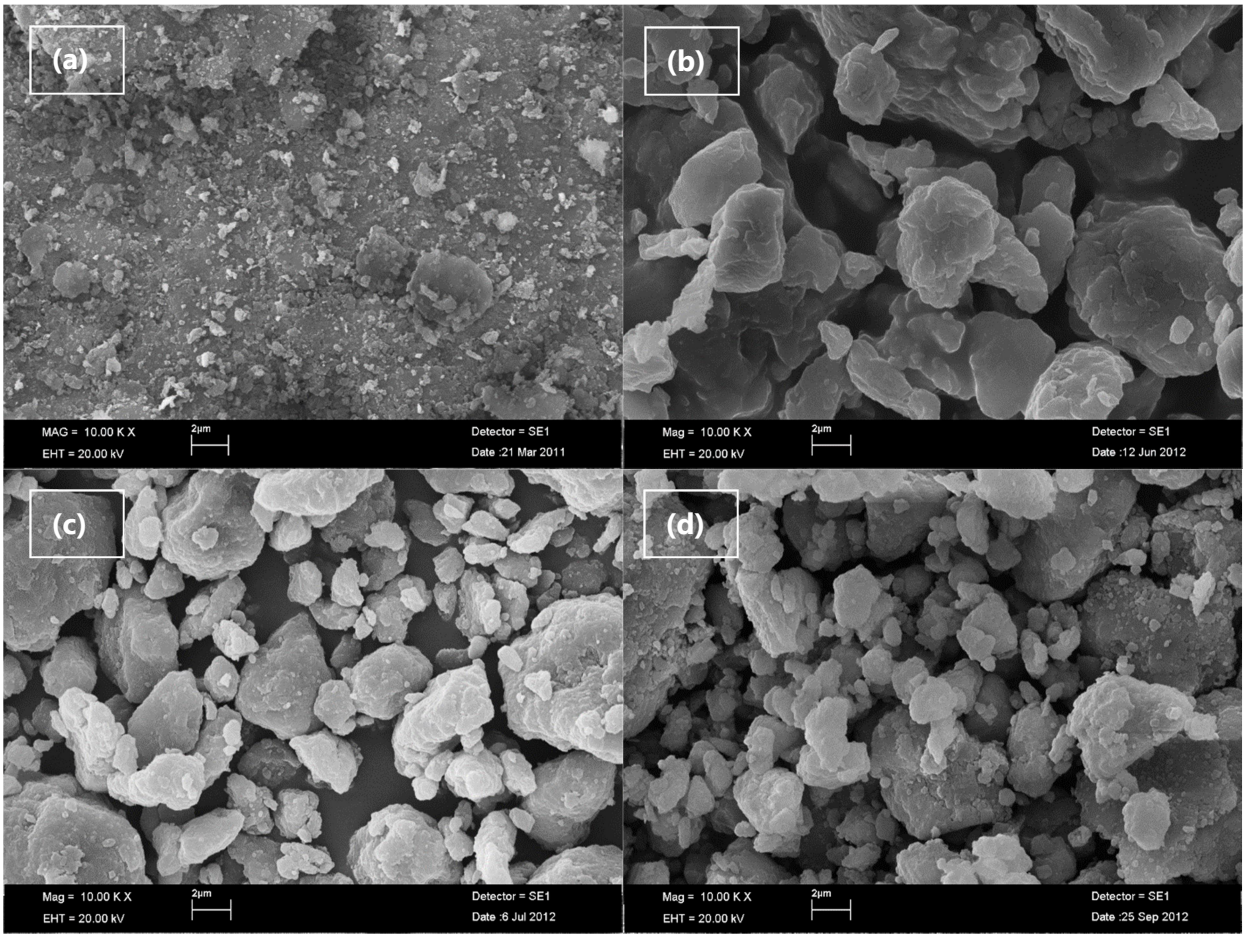


Figure 4. SEM Images of the Al- 10 % Al_2O_3 Powder Mixtures Milled for (a) 30 Hours, (b) 60 Hours, (c) 90 Hours and (d) 120 Hours.

Figure 5 and Figure 6 show microstructures of Al-% 15 Al_2O_3 and Al-% 20 Al_2O_3 powder mixtures milled for different periods, respectively. For the powder mixture of Al-% 15 Al_2O_3 flattening tendency which starts at the initial stages of milling can still be observed after 60 hours of milling. After 90 hours of milling it can be seen that flattening tendency ends and spherical particles can be observed.

When SEM images of Al-% 20 Al_2O_3 specimen is investigated it can be seen that formation of small particles starts earlier because of the increasing Al_2O_3 content.

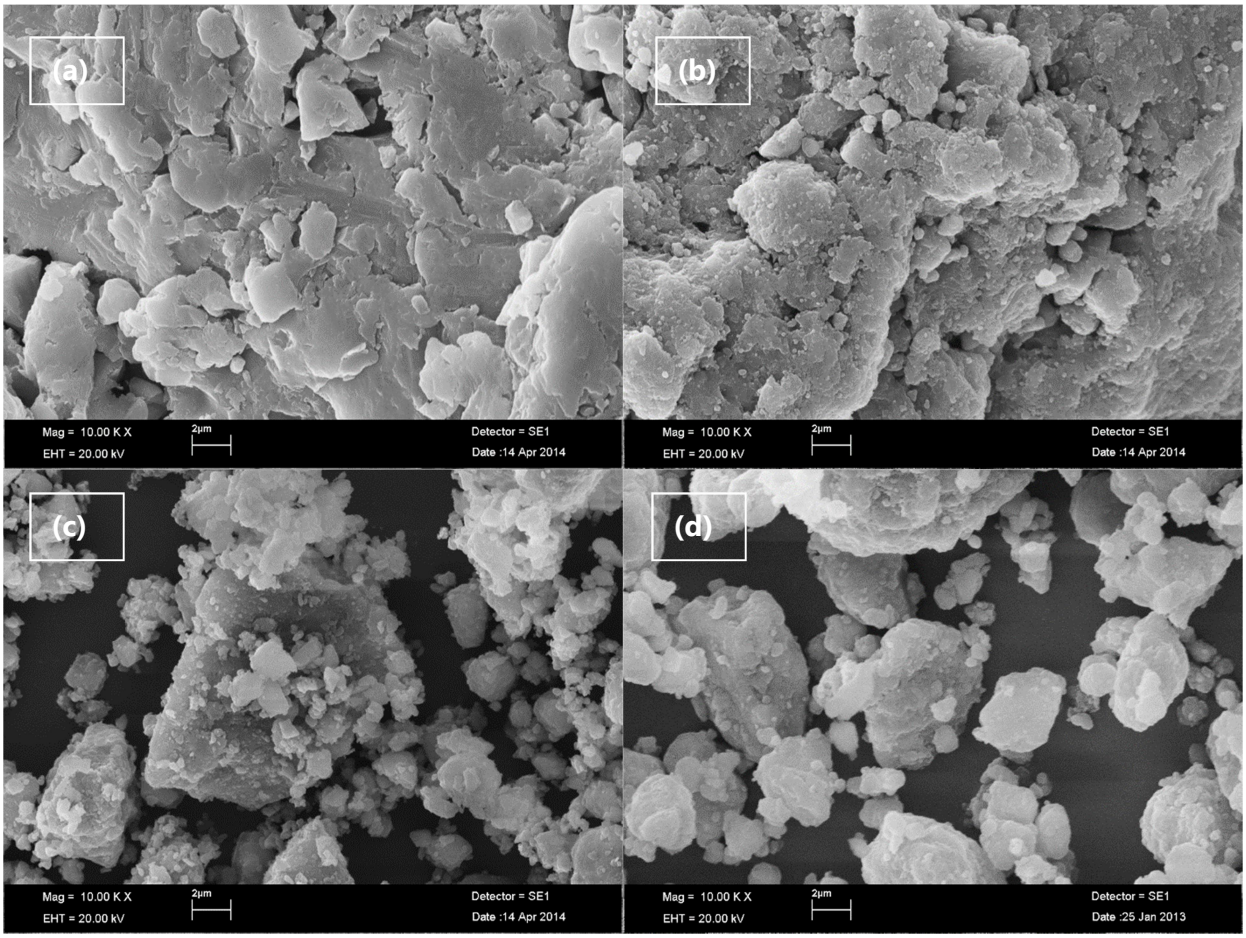
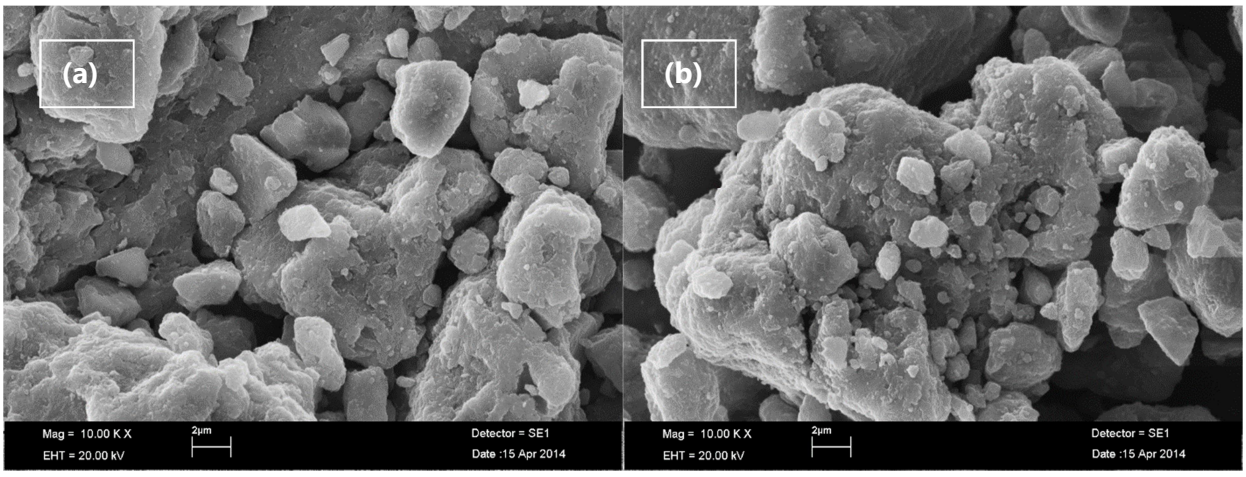


Figure 5. SEM Images of the Al- 15 % Al_2O_3 Powder Mixtures Milled for (a) 30 Hours, (b) 60 Hours, (c) 90 Hours and (d) 120 Hours.

When the 120 hour milled microstructures of the samples that contain different weight ratio of reinforcing element are compared, it can be easily said that number of spherical particles increase by increasing Al_2O_3 content.



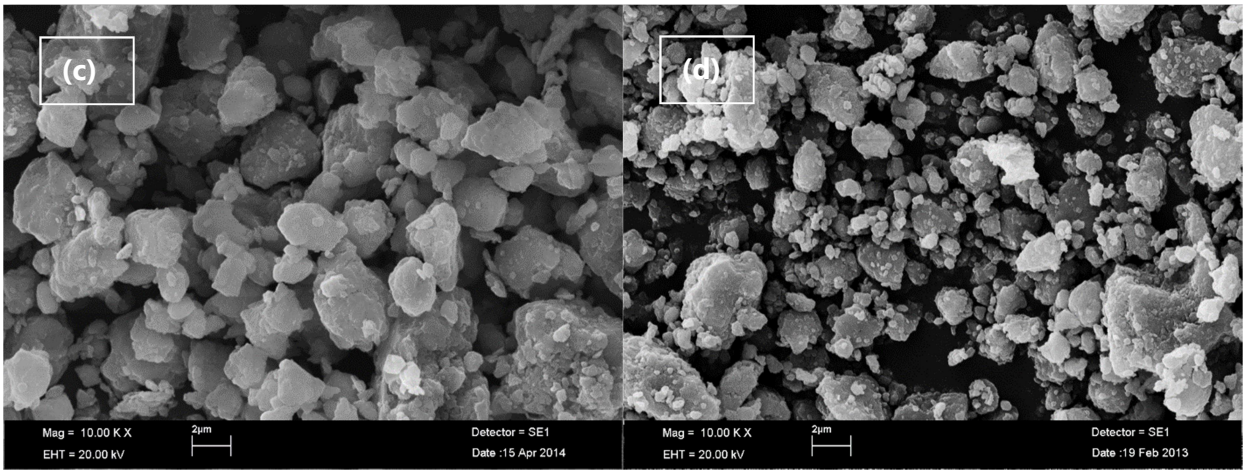


Figure 6. SEM Images of the Al- 20 % Al_2O_3 Powder Mixtures Milled for (a) 30 Hours, (b) 60 Hours, (c) 90 Hours and (d) 120 Hours

While evaluating the particle size change of specimens during milling, 12 images taken at six different magnitudes of 100X, 500X, 1 KX, 2 KX, 5 KX, 10 KX and 20 KX were processed by Image ProPlus software for each specimen. Figure 7 gives an example for the analyze.

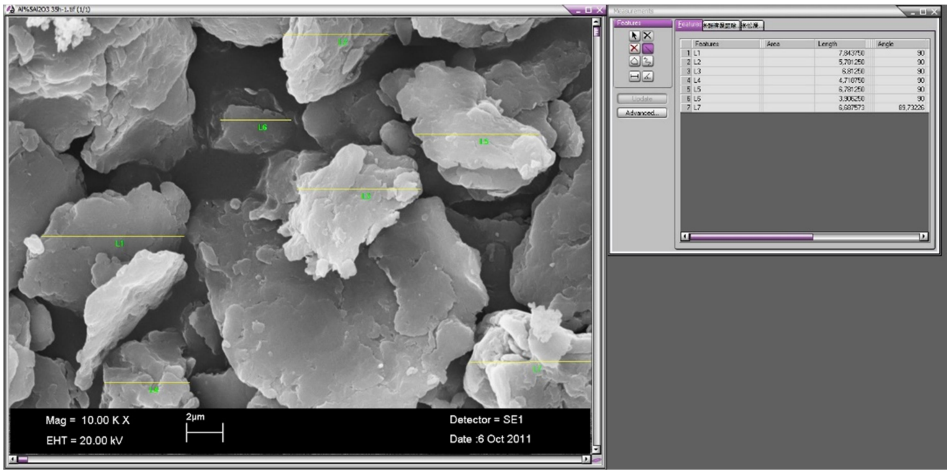


Figure 7. Average Particle Size Measuring Example of 30 Hours Milled Al- 5 % SiC Powder Mixture by Image ProPlus ®.

Figure 8 and Table 3 give the particle size change vs milling time. As expected particle size of each specimen decrease as milling process goes further. After early stages of milling average particle size of the samples changes in a wide range between 6,257 – 120,06 µm. That significant result may be due to the dominant cold welding mechanism during milling. As milling goes further to 60 hours same trend still goes on. But at the progressive stages, because a steady state is reached between cold welding and fracturing mechanisms, particle sizes of the specimens became steady. So final particle sizes of the samples are close to each other.

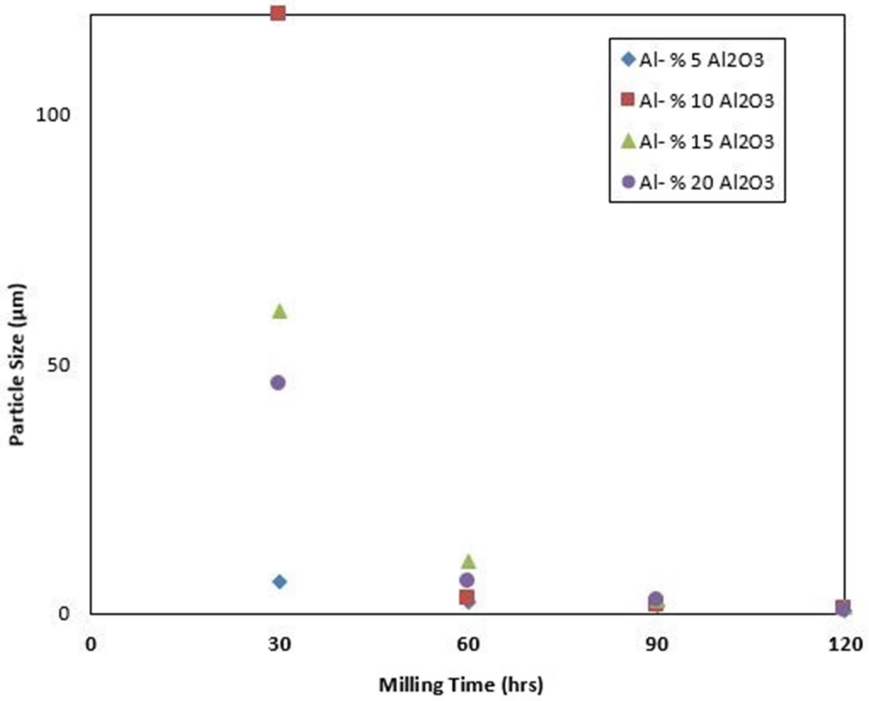


Figure 8. Particle Size Change of Al-Al_2O_3 Composite Powder Mixtures by Milling Time.

Table 3. Particle Size Change against Milling Time.

Milling Time (Hour)	Particle Size of Al-Al_2O_3 Powder Particles After Milling (µm)			
	Al_2O_3 Content (% Wt.)			
	5 %	**10 %**	**15 %**	**20 %**
30	6,257	120,06	60,795	46,035
60	2,280	2,775	10,485	6,347
90	1,544	1,653	2,610	2,701
120	0,696	0,976	1,078	0,512

3.2. XRD Results

Figure 9 gives the XRD pattern of as-received Al and Al_2O_3. In order to investigate if any contamination exists because of the stearic acid added XRD pattern of the stearic acid was also taken.

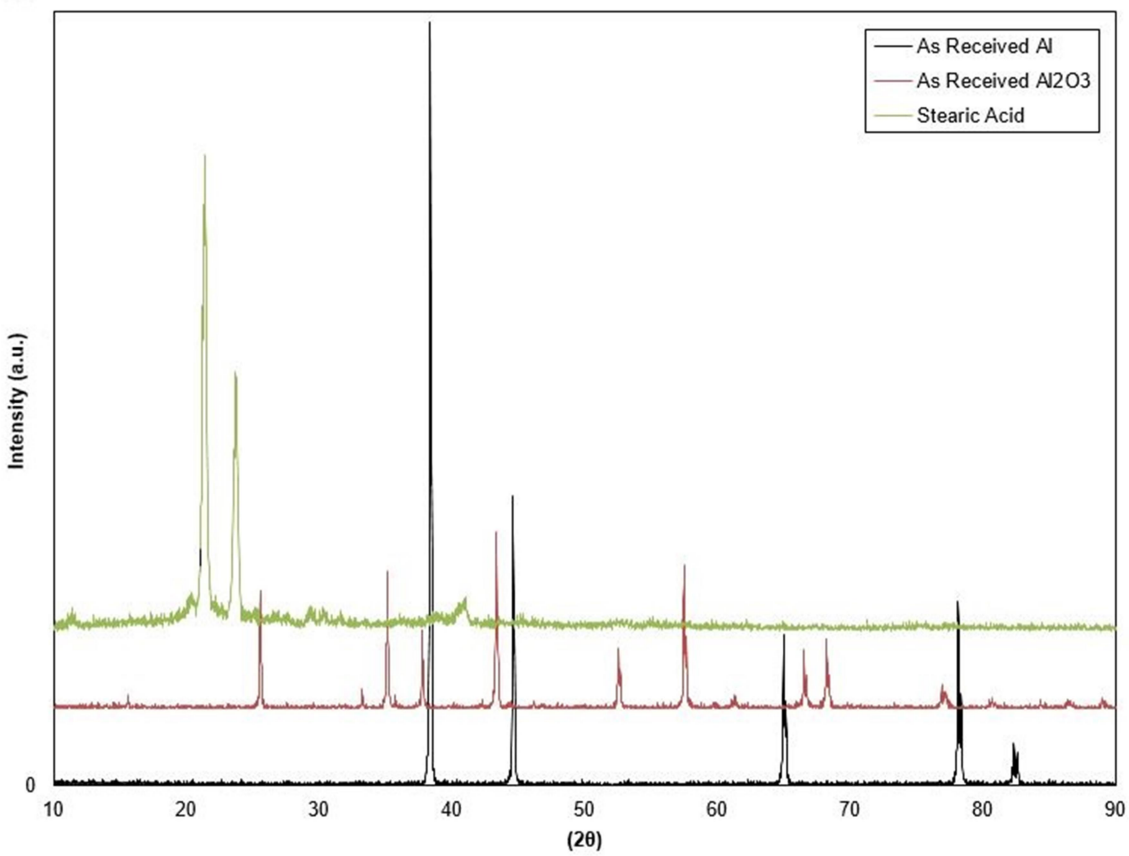


Figure 9. XRD patterns of the as received pure Al, Al_2O_3 and Stearic Acid.

From Figure 10, it is obvious that because of the high Al content of the mixture, all major peaks observed in the pattern match pure Al peaks recorded. But also intensity of those peaks decreases by milling time. For example the intensity of the major Al peak recorded at 2θ=38,38° decreases to 104 after 120 hours of milling. And XRD patterns also show that 2θ degrees increase by increasing milling time. According to the Bragg's Law that result shows the distance between atomic planes decrease by the milling process.

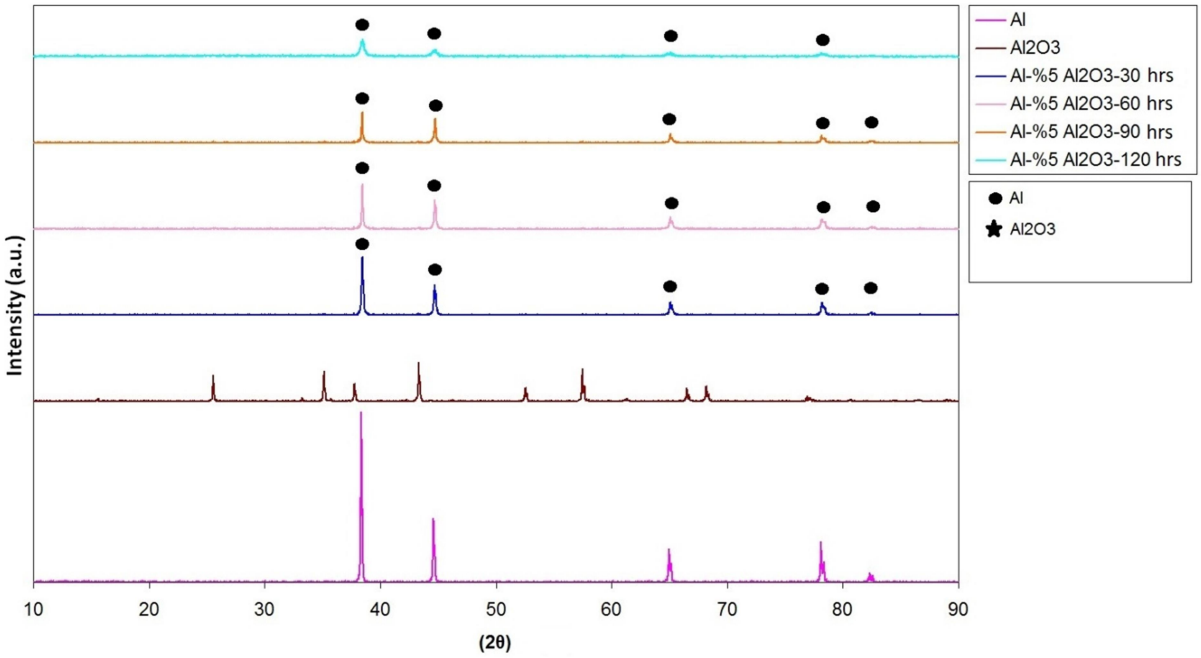


Figure 10. XRD patterns of the Al- 5 % Al_2O_3 Powder Mixtures Milled for 30 Hours, 60 Hours, 90 Hours and 120 Hours.

Figure 11 gives the XRD powder pattern for Al- % 10 Al_2O_3 mixture. Although similar results to the Al- % 5 Al_2O_3 can be said, due to the increasing Al_2O_3 content pure Al_2O_3 peaks like the one at 2θ = 35,14° can be seen initially. But the intensity of that peak decreases and then disappears by increasing milling time.

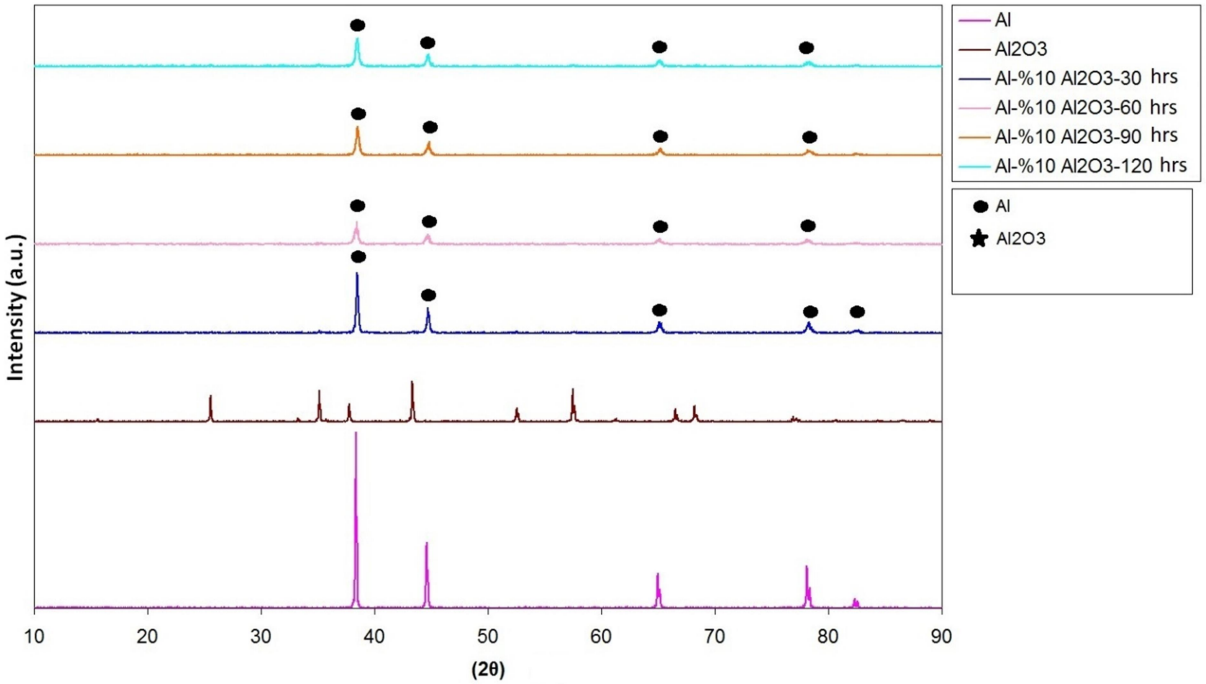


Figure 11. XRD patterns of the Al- 10 % Al_2O_3 Powder Mixtures Milled for 30 Hours, 60 Hours, 90 Hours and 120 Hours.

Al- % 15 Al_2O_3 X-RD pattern can be seen in Figure 12. Significant peaks that appear at 2θ degrees 43,34°, 52,68° and 57,44° can be seen for the first time and belong to pure Al_2O_3. Similar results and peaks can be observed due to Al_2O_3 content at Al- % 20 Al_2O_3 XRD results (Figure 13).

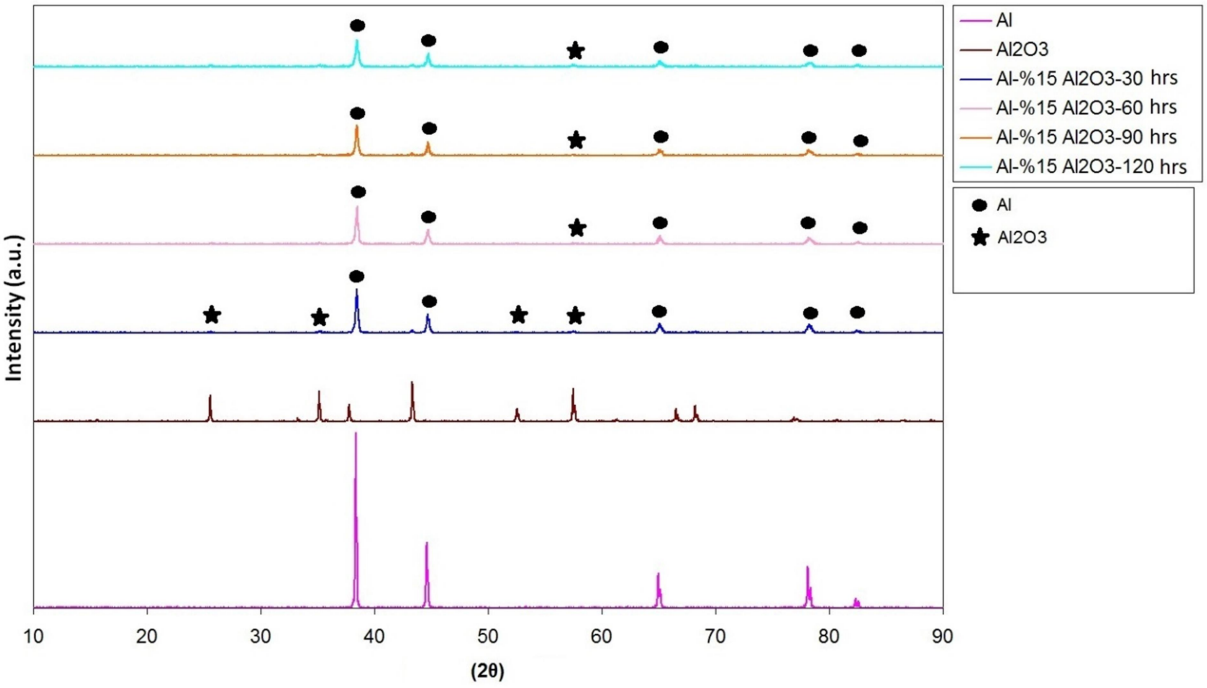


Figure 12. XRD patterns of the Al- 15 % Al_2O_3 Powder Mixtures Milled for 30 Hours, 60 Hours, 90 Hours and 120 Hours.

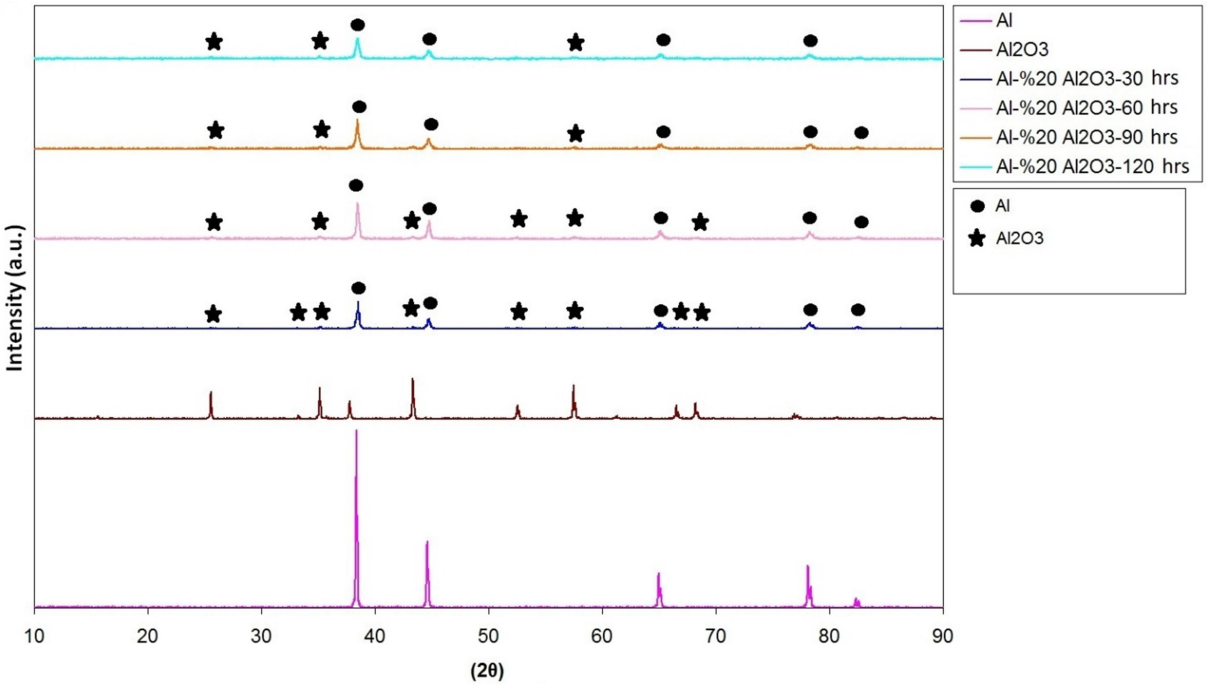


Figure 13. XRD patterns of the Al- 20 % Al_2O_3 Powder Mixtures Milled for 30 Hours, 60 Hours, 90 Hours and 120 Hours.

3.3. BET Surface Area and Pore Size Results

Table 4 gives single point, BET surface area and pore size results for 120 hour milled Al and Al_2O_3 composite powders. Figure 14 summarizes the data graphically.

Table 4. BET, Single Point Surface Area and Pore Size Results.

	Single Point Surface Area (m^2/g)	BET Surface Area (m^2/g)	Pore Size (nm)
Al	0,228	0,092	51,994
Al_2O_3	0,199	0,086	45,441
Al-% 5 Al_20_3	6,184	4,245	26,888
Al-% 10 Al_20_3	4,659	1,438	61,720
Al-% 15 Al_20_3	4,336	2,820	33,827
Al-% 20 Al_20_3	4,140	3,414	28,113

As can be seen BET and single surface area values are higher than those of as-received Al and Al_2O_3. That significant change in surface area can be associated with decreasing particle size. Generally single point surface area results of the 120 hour milled specimens are close to each other.

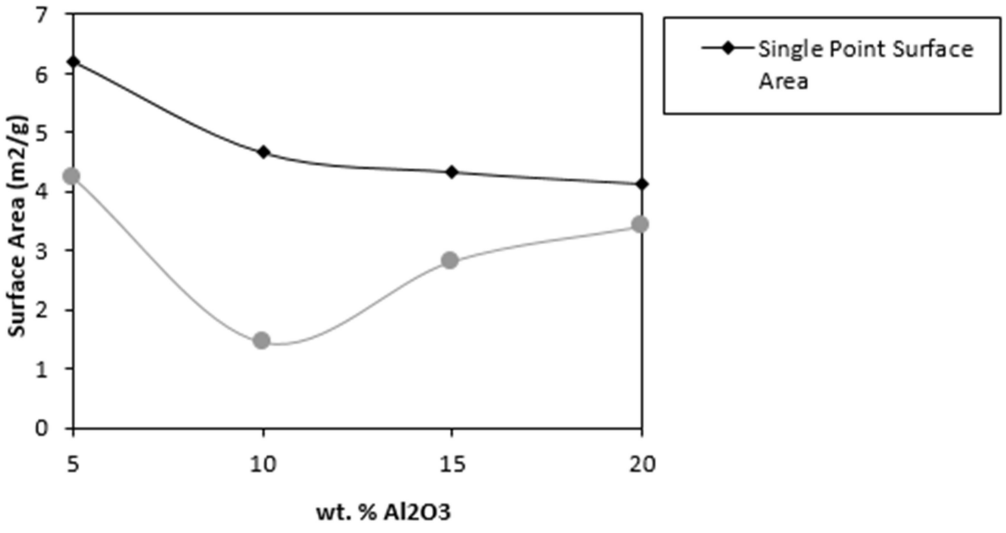


Figure 14. Single and Bet Surface Area Change by Weight Ratio of Al_2O_3.

The highest BET surface area value belongs to Al-% 5 Al_2O_3 composite mixture in accordance with the lowest pore size value (Figure 15).

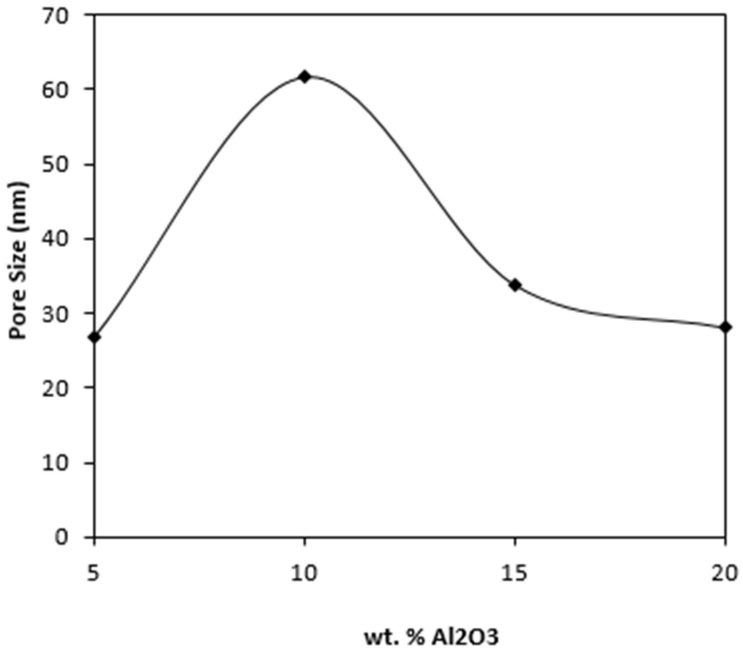


Figure 15. Pore Size Change by Weight Ratio of Al_2O_3.

Acknowledgements

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References

1. Suryanarayana, C. 2001. Mechanical alloying and milling. Progress in Materials Science, 46:1-184.
2. Suryanarayana, C., 2008. Recent developments in mechanical alloying. Reviews on Advanced Materials Science, 18:203-211.
3. Jiang, X., Trunov, M.A., Schoenitz, M., Dave, R.N., Dreizin, E.L., 2009. Mechanical alloying and reactive milling in a high energy planetary mill. Journal of Alloys and Compounds 478:246-251.
4. Zhang, B.Q. Lu, L., ,Lai, M.O. 2003. Physica B, 325:120–129.
5. Sivasankaran, S., Sivasprasad, K., Narayanasamy, R., Kumar Iyer, V., 2010. An investigation on flowability and compressibility of AA 6061100 − x-x wt.% TiO2 micro and nanocomposite powder prepared by blending and mechanical alloying. Powder Technology 201:70-82.
6. Smagorinski, M.E., Tsantrizos, P.G., Grenier, S., Cavasin, A., Brzezinski, T., Kim, G., 1998. The properties and microstructure of Al-based composites reinforced with ceramic particles, Materials Science and Engineering A 244 (1): 86–90.
7. Arik, H., 2008. Effect of mechanical alloying process on mechanical properties of α–Si3N4 reinforced aluminum based composite materials. Materials and Design 29: 1856-1861.
8. Tekmen C., Ozdemir, I., Fritsche, G., Tsunekawa, Y., 2009. Structural evolution of mechanically alloyed Al–12Si/TiB2/h-BN composite powder coating by atmospheric plasma spraying. Surface & Coatings Technology 203:2046-2051.

9. Tavoosi, M., Karimzadeh, F., Enayati, M.H., Heidarpour, A., 2009. Bulk Al–Zn/Al2O3 nanocomposite prepared by reactive milling and hot pressing methods. Journal of Alloys and Compounds 475:198-201.
10. Cambronero, L.E.G., Sánchez, E., Ruiz-Roman, J.M., Ruiz-Prieto, J.M., 2003. Mechanical characterisation of AA7015 aluminium alloy reinforced with ceramics, Journal of Materials Processing Technology 143–144: 378–383.
11. Prabhu B., Suryanarayana C., An L., Vaidyanathan R., 2006. Synthesis and characterization of high volume fraction Al-Al_20_3 nanocomosite powders by high-energy milling. Materials Science and Engineering A, 425:192-200.
12. Wan-Li, G., 2006. Bulk Al-SiC nanocomposite prepared by ball milling and hot pressing method. Trans. Nonferrous Met. SOC. China 16:398-401.
13. Nie, C., Gu, J., Liu, J., Zhang, D., 2008. Investigation on microstructures and interface character of B4C particles reinforced 2024Al matrix composites fabricated by mechanical alloying. Journal of Alloys and Compounds 454:118-122.
14. Kubota, M., 2010. Solid state reaction in mechanically milled and spark plasma sinteren Al-B_4C composite materials. Journal Of Alloys And Compounds, 504: 319-322.
15. Wang, J., 2008. Mechanical alloying of amorphous Al–SiO_2 powders. Journal of Alloys and Compounds 456:139-142.