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Effect of Stirring Time and Processing Temperature on the Size Distribution of Al6061-Al_2O_3 Powder Mixture Produced by In Situ Powder Metallurgy (IPM) Method

Farshad Akhlaghi[1], Saeed Pour-Naderi[2]

[1]School of Metallurgy and Materials Engineering, Faculty of Engineering, University of Tehran
P.O.Box 11155-4563, Tehran, Iran
[2]School of Metallurgy and Materials Engineering, Faculty of Engineering, University of Tehran
P.O.Box 11155-4563, Tehran, Iran

A relatively new technique termed In situ Powder Metallurgy (IPM) was utilized for processing of Al6061-Al_2O_3 powder mixtures. In this technique a mixture of specified amounts of alumina particles and aluminum alloy melt, is stirred via an impeller at a certain temperature for a predetermined time. The kinetic energy of the impeller is transferred to the melt via the non-wetting alumina particles and results in melt disintegration. The liquid droplets created by this process are then solidified upon cooling the blend resulting in a mixture of Al alloy powders and alumina particles. This mixture can be subsequently used as a feedstock for preparation of Al-Al_2O_3 composites via the standard powder metallurgy methods. In the present study the effect of stirring time (4-10 min) and temperature (710 and 780 °C) on the size distribution of the powder mixture is investigated. It is concluded that the morphology of Al powders is not affected by the processing time or temperature and the finest powder particles are produced after stirring at temperatures of 710 °C for 8 minutes.

Keywords: *Al6061-Al_2O_3 composite; In situ Powder Metallurgy method; Stirring time; Melt temperature.*

1. Introduction

A l-Al_2O_3 composites can be regarded as an important group of Al-based metal matrix composites (Al-MMCs) which are being used in the aerospace and automobile industries. These materials have received attention due to their improved mechanical [1], tribological [2,3] and high temperature properties [4,5]. They are produced by several methods such as casting [6], infiltration [7,8], in situ [9] and powder metallurgy (PM) [10,11]. MMCs produced by PM routes exhibit a more uniform distribution of the second phase within the matrix material as well as less degradation of the reinforcement due to their lower processing temperatures as compared with the casting methods. However, metallic powders are expensive and a limited number of Al alloy powders are commercially available. In order to overcome these problems, a new processing route termed In situ Powder Metallurgy (IPM) method has been developed in which the bulk Al alloy is used as the starting material. This method combines the advantages of powder metallurgy (P/M) with those of the stir casting, but avoids at the same time their respective drawbacks. The IPM method has been successfully used to produce Al/Graphite composites [12,13].

In the present study, for the first time the IPM method is used for processing of Al/Al_2O_3 powder mixtures. The size distribution and morphology of the powders are two important factors that influence the compaction behavior, distribution of the Al_2O_3 particles within the matrix and properties of the consolidated Al/Al_2O_3 composites. In this paper we aim to gain a better understanding of the IPM process through investigating the effect of stirring time (in the range of 2-10 min) at two different processing temperatures of 710 °C and 780 °C on the morphology and size distribution of the produced powder mixtures.

2. Process Description

The present process utilizes the non-wettability of alumina particles with molten aluminum alloy together with the shear forces induced by the impeller for melt disintegration. The melt disintegration occurs by kinetic energy transfer from the stirrer to the metal, via a solid media (alumina particles) for break up a liquid pool into droplets. In fact, during vigorous and relatively high speed stirring of the suspension of non-wetting alumina particles in metallic melts, some of the binding forces between atoms which hold a mass of liquid metal together are broken. This process results in the formation of gas bridges within the mass of molten metal and creation of discrete droplets as shown schematically in Fig. 1-(a). The role of the alumina particles is to separate these molten droplets and prevent their adjoining. These still molten metal pieces, therefore, disintegrate into finer droplets as long as the shear force induced by stirring exceeds the cohesive forces due to surface tension that act to preserve their integrity (Fig. 1-(b)). As droplets get progressively smaller through disintegration, to a critical size, the surface tension forces that are inversely proportional to diameter become sufficiently large to resist the shear forces responsible for the break up. At this point, melt break up ceases. However, during stirring, the small molten metal pieces can also be subjected to several coalescence intercourses that lead to their re-unification. The liquid droplets created by this process are then solidified upon cooling the blend resulting in a mixture of alloy powders and non-wetting particles (Fig. 1-(c)). At this stage, the surface tension forces tend to spherodize the irregular liquid droplets provided that they have not yet cooled to a point where the viscosity has greatly increased or they have already solidified. This mixture can be subsequently used as a feedstock for preparation of composites via the standard powder metallurgy methods.

This method is basically similar to another process termed "Solid-Assisted Melt Disintegration (SAMD)" which has been developed for the preparation of aluminum alloy powder particles [14-16]. In the SAMD process, melt disintegration is achieved by introducing a solid media (i.e. NaCl or alumina) into the molten alloy and stirring the slurry to produce droplets which form the powder particles after solidification. The resulting mixture of aluminum powders and solid media particles is subjected to water dissolution or sieving to separate NaCl or alumina from the aluminum powder particles. However, In the IPM method the solid medium (i.e. alumina particles) is not separated from the alloy powders but remains in the mixture to act as the reinforcing phase in the final consolidated composite.

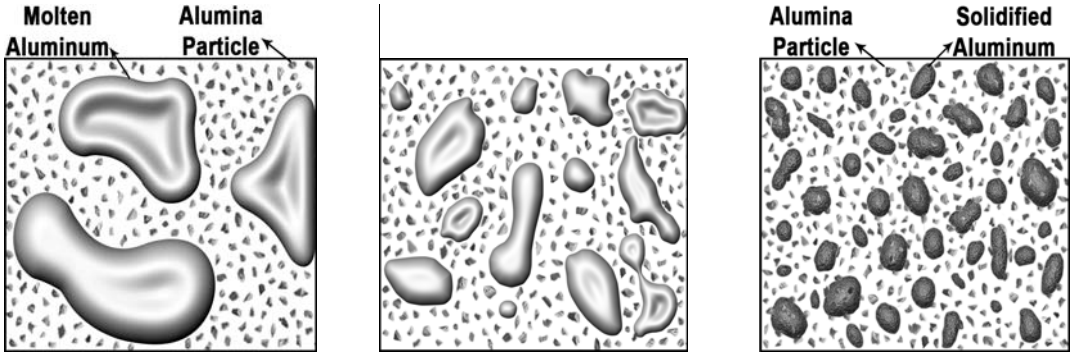


Fig. 1. Schematic illustration showing the generation of Al powders distributed within alumina particles in the IPM method, (a): generation of large Al droplets separated from each other by alumina particles at the initial stages of stirring, (b): disintegration of large droplets into smaller ones by further stirring and (c): formation of Al powders after solidification of droplets.

3. Materials and Experimental Procedures

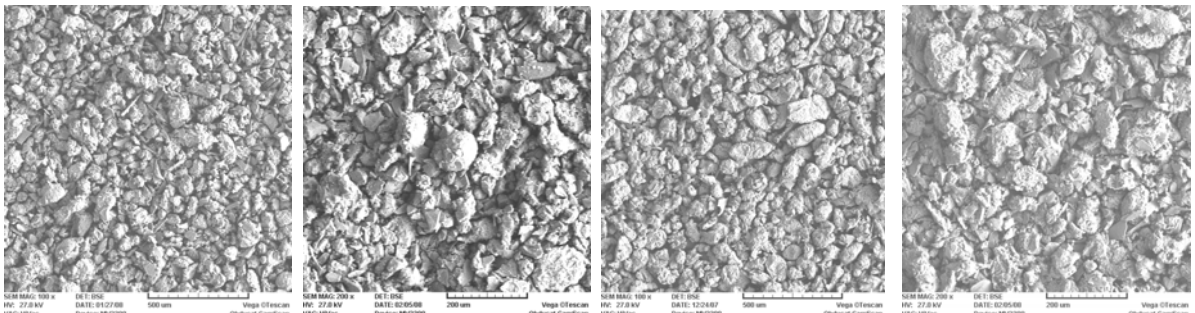
In this study, aluminum alloy 6061 of nominal compositions (in wt.%) of Al-1.12Mg, 0.11Mn, 0.072Zn, 0.04Cr, 0.33Cu, 0.48Fe, 0.64Si was used for making the matrix alloy powders while

α-Al_2O_3 (alumina) particles having the size of <38μm, were used as the reinforcing phase. About 50g of aluminum alloy together with 50g of alumina particles were charged in a clay-bonded graphite crucible of 0.5-Kg capacity and the alloy was melted using an electrical resistance-heated laboratory furnace. The temperature of the alloy was raised to about 710 °C or 780 °C and the mixture was stirred for different periods of time ranging from 2 to 10 min at 1400 rpm. A spiral graphite impeller was used for stirring. This impeller was preheated to 500 °C prior to each experiment. The blend was then quenched in water and the resultant Al+ alumina powder mixture was dried and subjected to sieve analysis. The particles having the size of <500 μm were subjected to laser particle size analyzing. The shape and surface morphology of powders were examined by scanning electron microscopy (SEM).

4. Results and discussion

4.1 Morphological analysis

Fig. 2 shows the typical low and high magnification SEM micrographs of the mixture of aluminum and alumina powder particles processed at two different temperatures of 710 and 780 °C. The low magnification micrographs reveal the presence of free Al_2O_3 particles distributed uniformly within the Al powder particles. It can be seen that the morphology of Al powders is quasi-spherical and has not been affected by the processing temperature. The high magnification SEM micrographs show that some alumina particles have been attached to the surface of Al powders. It can be seen that the alumina particles were only attached to the relatively coarse (i.e. >250 μm) Al powders. This can be attributed to the higher heat content of the larger droplets which resulted in a higher liquid fraction at the conclusion of stirring. The solidification shrinkage of these droplets resulted in mechanical bonding of the fine Al_2O_3 particles within the surface cavities of these particles generated during their solidification. It must be noted that due to the different densities and flow characteristics of Al and Al_2O_3 particles, the attachment of these particles to each other can be regarded as a beneficial effect for obtaining a uniform distribution of the reinforcing particles within the matrix alloy in the final consolidated material.



(a) **(b)** **(c)** **(d)**

Fig. 2. Typical SEM micrographs of Al 6061+Al_2O_3 powder mixtures processed at different temperatures of (a , b):710 °C and (c , d): 780 °C.

4.2. Size distribution of particles

In the PM terminology, the particles having the size smaller than 1mm can be regarded as powders [17]. The results indicate that the powder mixtures prepared in the present work have a wide size distribution ranging from 10 to 900 μm. However, Al particles having the size larger than 500μm contained flakes and in some cases exhibited very large sizes indicating insufficient energy induced to the melt during melt disintegration. Therefore, we considered only the proportion of powder of less than 500μm particle size as the useful material, i.e. the yield of the powder. Fig. 3(a,b) are typical

particle size distribution plots obtained from laser particle size analysis of sub-500µm sized Al +Al_2O_3 powders produced at processing temperatures of 710 and 780 °C. By considering such plots it was concluded that by using the present set of experimental conditions, the particle size distributions were bi-modal with the dominant mode on the small particle side. This bi-modal distribution can be explained by the mechanisms controlling the disintegration and re-unification of droplets during the process as was mentioned before. In addition, not a considerable sizes smaller than 10 µm can be detected in the powders.

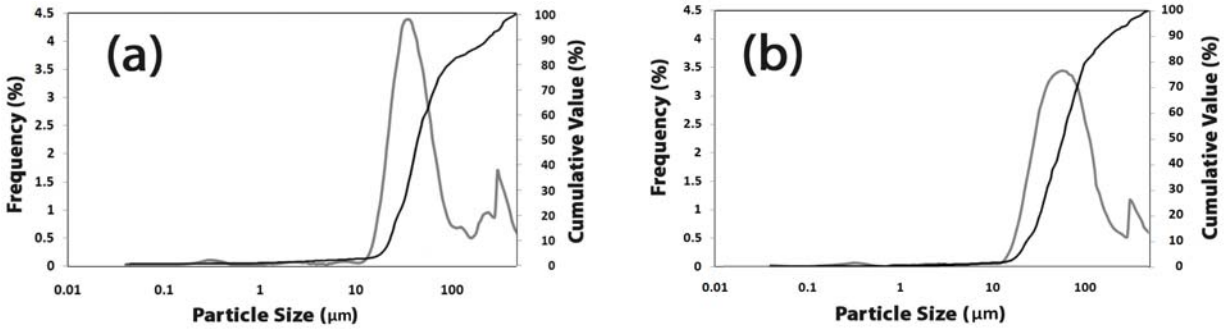


Fig. 3. Typical particle size distribution plots obtained from laser particle size analysis of sub-500µm sized Al +Al_2O_3 Powders produced at different processing temperatures of (a): 710 and (b)780 °C.

The variation of the yield of the Al+Al_2O_3 powder mixtures under500µm as a function of stirring time for powders produced at two different temperatures of 710 and 780 °C is shown in Fig. 4. It can be seen that stirring for 4 min resulted in a relatively low yield for both of the processing temperatures. However, by increasing the duration of stirring to 6 min, the yield has increased. The yield of the powder has not changed considerably by further increasing the stirring time to 10 min at 710 °C, but the yield decreased for 780 °C. These results can be explained by disintegration and adjoining events that may take place simultaneously during stirring as was mentioned before. In fact, with increased temperature and fluidity of the melt, the available energy can be effectively utilized in disintegrating the melt, giving finer droplets. However, according to the present results, the lower fluidity of the melt at 710 °C results in a higher yield of <500 µm particles. In fact, during stirring, the small molten metal pieces can also be subjected to several coalescence intercourses that lead to their reunification. In addition, in the time interval between the conclusion of stirring and completion of the melt solidification, the molten droplets can flow between the solid medium (Al_2O_3) particles due to gravity and join together leading to coarser droplets. It is obvious that this effect is intensified for the melts having a higher fluidity. It seems that the last two mentioned mechanisms for enhanced re-unification of more fluid droplets are dominant over their increased disintegration. Therefore, the increased fluidity of the melt resulted in formation of a higher amount of coarse (i.e. >500µm) powder particles.

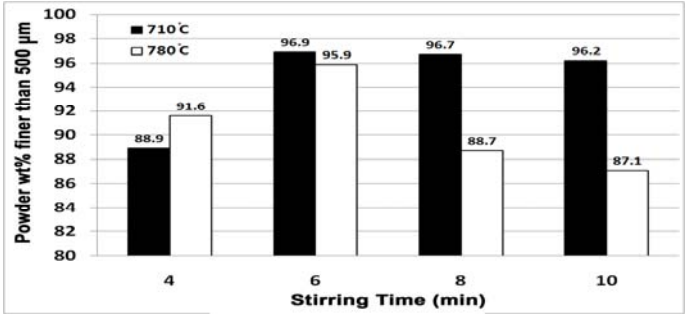


Fig. 4. The variation of the yield of the Al+Al_2O_3 powder mixtures under 500µm as a function of stirring time for powders produced at two different temperatures of 710 and 780 °C.

The variation of D_{10}, D_{50} and D_{90} of the powder mixtures as a function of stirring time were obtained from the cumulative size distribution curves (such as those shown in Fig.3) and are shown in Fig. 5(a-c). It can be seen that stirring time had little effect on the size of the particles in the finest size range (i.e. D_{10}), but at the lower temperature used (710 °C) the powder became finer. However, this size difference is diminished after 10 min stirring. Similar trend is observed for the median particle size (D_{50}) as shown in Fig. 5-b. The weak dependence of D_{10} and D_{50} on the stirring time can be explained by this fact that the average size of alumina particles was less than 38μm and therefore the majority of these particles were accumulated in the fine size range (i.e.<70μm) of the particles. Laser particle size analyzing conducted on the as-received and stirred alumina particles exhibited identical results confirming that the alumina particles do not break or agglomerate during the process so that the increased stirring time does not affect the size of alumina particles. Therefore, the presence of a high amount of these solid particles in the smaller size bands of the powder mixture resulted in a weak dependence of D_{10} and D_{50} on the stirring time. However, as shown in Fig.5-c, the D_{90} values have been affected by the stirring time at least for the times in excess of 6 min. In fact according to Fig. 4, after 6 min of stirring, more than 90% of the aluminum was converted to powder particles having the size of <500μm irrespective of the processing temperature. The increased stirring time at 780 °C resulted in creation of more particles in the size range of <150μm after 10 min of stirring (Fig. 5-c) and at the same time re-unification of larger droplets and formation of a high fraction of >500 μm particles due to a relatively high fluidity of the molten aluminum. In case of stirring at 710 °C, due to a higher content of <500 μm droplets in the mixture (as compared with 780 °C), when the stirring time exceeds an optimum value of 8 min, the re-unification phenomenon is intensified resulting in increased D_{90} of the particle size distribution.

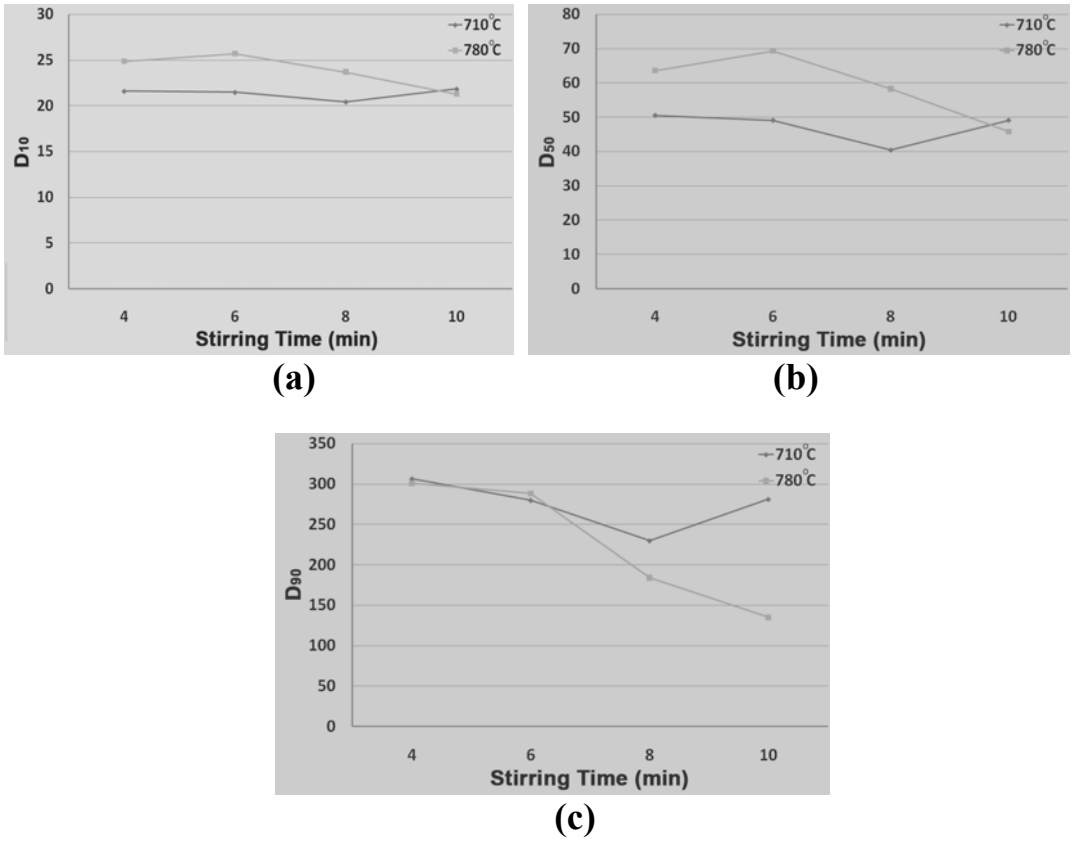


Fig. 5. The variation of (a) D_{10}, (b) D_{50} and (c) D_{90} of the powder mixtures as a function of stirring time.

5. Conclusions

1-The In situ Powder Metallurgy (IPM) technique demonstrated the capability to produce a mixture of alumina and alumnium 6061 powder particles by using alumina powers and aluminum alloy ingots as the starting materials. The produced powders can be used as a feedstock for preparation of Al-Al_2O_3 composites.

2- SEM micrographs of the mixture of aluminum and alumina powder particles revealed the presence of free Al_2O_3 particles distributed uniformly within the Al powder particles. The morphology of Al powders was quasi-spherical and was not affected by the processing time or temperature. The high magnification SEM micrographs showed that some alumina particles were attached to the surface of the relatively coarse Al powders.

3- The yield of sub-500μm particles was affected by the stirring time and processing temperature and exhibited its maximum value (i.e.> 96.5%) for the powder particles processed at 710 °C and stirred for 6-8 minutes. These results were explained in terms of the melt disintegration and droplet adjoining events that could take place simultaneously during stirring.

4- The size distributions of the sub-500 μm Al+ Al_2O_3 powder mixtures were always bi-modal with the dominant mode on the small particle size.

5- Neither the stirring time in the range of 4 to 10 min nor the processing temperature (710 or 780 °C) did not show any considerable effect on the D_{10} and D_{50} values of the sub-500 μm Al+ Al_2O_3 powder mixtures. However, the D_{90} values decreased with increasing stirring time and exhibited lower values for the powders processed at the higher temperature.

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