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Research Article

Effects of Al_2O_3 Particulate Addition on Mechanical Properties of Vinyl Ester Matrix Composite Material

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Abstract

Vinyl ester resins are widely used as matrix materials in particle and fiber reinforced composites. Nowadays, particles are also preferred in micron-sized microstructures to increase the mechanical properties of polymer matrix composites. In this work, vinyl ester matrix composites reinforced with aluminum oxide particles of 1-3 micron dimensions were produced. Tensile, hardness, bending, wear, impact strength properties of the specimens prepared at different ratios were investigated. Teflon mold was preferred in the preparation of test samples. 50% active methyl ethyl ketone peroxide as initiator and 6% cobalt naphthenate as accelerator were used in the reaction. From 0.5% to 2% by weight of aluminum oxide was added to the vinyl ester resin. In the ultrasonic mixer, the formulation was stirred for 30 minutes by adding MEG (mono ethylene glycol) to 5 times the amount of particulate in order to ensure the distribution of the particle in the mixture and to prevent agglomeration. The tensile and % elongation, impact, wear strength and hardness values increased by 1,5% by weight. A decrease in bending strength was observed.

Keywords: Aluminum oxide (Al_2O_3), Vinyl ester, Composite material, Mechanical properties

Al_2O_3 Partikül İlavesinin Vinil Ester Matrisli Kompozit Malzemenin Mekanik Özellikleri Üzerine Etkileri

Öz

Vinil ester reçineleri, partikül ve fiber takviyeli kompozitlerde matris malzemeleri olarak yaygın olarak kullanılmaktadır. Günümüzde polimer matrisli kompozitlerin mekanik özelliklerini artırmak için mikron boyutlu mikro yapılarda da partiküller tercih edilmektedir. Bu çalışmada 1-3 mikron boyutlarında alüminyum oksit partikülleri ile takviyelendirilmiş vinil ester matrisli kompozitler üretilmiştir. Farklı oranlarda hazırlanan numunelerin çekme, sertlik, eğilme, aşınma ve darbe dayanımı özellikleri incelenmiştir. Test numunelerinin hazırlanmasında teflon kalıp tercih edilmiştir. Reaksiyonda başlatıcı olarak %50 aktif metil etil keton peroksit ve hızlandırıcı olarak %6 kobalt naftenat kullanılmıştır. Vinil ester reçinesi içerisine ağırlıkça % 0,5'den % 2'ye kadar alüminyum oksit ilave edildi. Partikülün karışımdaki dağılımını sağlamak ve topaklaşmayı önlemek için partikül miktarının 5 katı MEG (mono etilen glikol) eklenerek formülasyon ultrasonik homojenizatörde 30 dakika karıştırılmıştır. Çekme ve % uzama, darbe, aşınma mukavemeti ve sertlik değerleri ağırlıkça %1,5 oranına kadar arttı. Eğilme dayanımında bir azalma gözlendi.

Anahtar Kelimeler: Alüminyum oksit (Al_2O_3), Vinil ester, Kompozit malzeme, Mekanik özellikler

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1. Introduction

Composites have become the dominant substitute for metals and other engineering materials in nearly all engineering applications. The reason behind the enormous growth of composites is the wide range of applications they have found themselves in. The most widely used type of composites is polymer matrix composites. Polymeric matrix composites are lightweight, economical, have low production costs, are resistant to corrosion and have the ability to produce parts with complex shapes. They are increasingly used in automotive, aerospace, defense, construction and many other commercial applications [1].

The mechanical properties of a polymer matrix composite can be improved by the addition of particle reinforcements. These properties can be designed by selecting the appropriate matrix and reinforcement material [2]. In general, the mechanical properties of particle-reinforced polymer composites depend on the shape, size and distribution of the reinforcement, as well as the adhesion between the matrix and the particles [3].

In addition to fiber reinforced composites, particle reinforced composites are also widely used. Particles can be used to increase ductility, reduce matrix modulus, and reduce the cost of composites [4-5]. Among the micro-scale particles used predominantly as filler/reinforcement, submicron particles have a much higher specific surface area. This high specific surface area drives the high surface interaction between the long-chain polymer matrices and the reinforcement. It is well known that the dispersion of submicron particles results in improved properties in polymer composites. If a homogeneous distribution of these submicron particles can be achieved, both the mechanical and electrical properties of the composite can be improved [6-8].

Modification of the polymer matrix is one of the approaches to developing new polymer materials. This modification can be done by adding different ceramic powders of different sizes to obtain the required mechanical properties [9]. Ceramics have high-temperature resistance and electrical insulation properties. Some ceramic materials have special properties such as magnetic, piezoelectric and superconductivity at very low temperatures. These properties are also preferred in composite materials [10]. There are many studies on the mechanical properties of particle-reinforced polymeric composites in the literature.

McGrath et al. [11] studied the effect of alumina powder in epoxy on mechanical properties. In this study, they found that the change in particle size, shape, and size distribution of alumina powder had little effect on the epoxy. However, resin crosslink density and filler loading were the most critical variables that could change all properties. Kardar et al. [12] studied the effect of nano alumina particles on the physical-mechanical properties of UV-cured epoxy acrylate via nano-indentation. In their studies, it was observed that the scratch resistance and self-healing of the film increased in the presence of nanoparticles. Hongxia Z. and Robert K.Y.Li [5] studied the effect of water absorption on the mechanical and dielectric properties of nano-alumina-filled epoxy nanocomposites. They found that mechanical properties such as the stiffness of the matrix increased with the addition of nano alumina, and also improved dielectric properties due to the increase in the total interfacial area. Zhao et al. [10] studied the mechanism leading to an improved mechanical performance in nanoscale alumina-filled epoxy. Their observations revealed that the improvement in tensile strength was due to the stronger interface.

Ramesh et al. [9] added different micro-modifier particles such as Al_2O_3, SiO_2 and TiO_2 separately at the rate of 10% into the glass fiber/epoxy structure and conducted a comparative study on the mechanical properties of the hybrid composites they produced. It was observed that the mechanical properties of all epoxy-modified composites were improved. It has been observed that Al_2O_3 particles in epoxy composites increase the hardness and impact energy compared to other ceramic particles. Iskender et al. [13] investigated the effect of micro and nanofiller content on the mechanical properties of epoxy composites. Micro-fillers are Al_2O_3, TiO_2 and fly ash added at rates between 10% and 30% by weight; Nanofillers are Al_2O_3, TiO_2 and clay, which are added at a rate of between 2.5% and 10% by weight. It was observed that the tensile strength, flexural strength and elongation at break values of the composites decreased, while the tensile modulus and bending modulus increased with increasing micro and nanofiller content ratio. Solmaz et al. [14] investigated the tensile properties and wear behavior of composite materials obtained by adding Al_2O_3 at different rates between 1% and 30% into the polyester matrix. They stated that the tensile strength and elongation amount decreased with the increase of Al_2O_3 ratio, and the wear resistance increased with the decrease in weight loss. Wenyan et al. [15] produced glass fiber reinforced vinyl ester resin composites modified with nano-Al_2O_3 by VARTM method. They modified the glass fiber surface with various contents of nano Al_2O_3 particles by the solid-liquid separation method. They examined the mechanical properties of the composite plates they obtained, and as a result of this experimental study, they determined that the tensile strength and bending strength increased with the increase of nano Al_2O_3 content, and the best mechanical performance was obtained when the nano Al_2O_3 ratio was between 0.5% and 1% by weight.

In this study, vinyl ester resin was preferred as the matrix material because it has properties between epoxies and unsaturated polyesters and is easy to use at room temperature. Vinyl ester resin has more hydroxyl terminal reactive double bonds than other resins. It has reactive double bonds only at the end of the chain, unlike unsaturated polyester resin, which has fully reactive double bonds in the chain. In vinyl ester resins, both the cure rate and the reaction conditions can be easily controlled. Vinyl ester resins are one of the widely used thermosets and provide excellent toughness, thermal stability and chemical resistance [16].

2. Material and Method

2.1. Materials

The vinyl ester resin (Erco Resin-Coating, Turkey) was used because it combines the properties of epoxies and unsaturated polyesters as matrix material and is easy to use at room temperature. Al_2O_3 particles were used with an average of 1-3 micron dimensions (PCT Plasma Ceramic Technology GmbH, powder specific surface area 55 ± 5 m^2/g, weight loss ≤ 2% after 2 hours at 700 ºC). Teflon was used as a mold material to prepare test samples. In the reaction, 50% active methyl ethyl ketone peroxide (MEK - Aldrich) was used as the initiator and 6% cobalt naphthenate (Aldrich) as the accelerator. MEG (mono ethylene glycol - Dow Chemical) was used to ensure a homogeneous distribution of the particles and to prevent agglomeration.

2.2. Preparing of Composite Specimens

The aluminum oxide was added (0.5, 1, 1.5, 2 % by weight) to the vinyl ester resin. Since the resin could not wet the aluminum oxide particles, it was not continued beyond 2%. In the sample preparation process, Aluminum oxide particles were mixed with an ultrasonic mixer for 30 minutes in MEG (mono ethylene glycol) at a ratio of 5 times the particle amount to ensure homogeneous distribution and prevent aggregation. Vinyl ester resin was placed in a container and aluminum oxide dispersed in MEG at the rate of 0.5% by weight of the total amount was added into the resin and mixing was applied with a mechanical mixer for 10 minutes. Following this process, 3% MEK peroxide was added as a hardener and mixed, then 1% cobalt naphthenate was added as an accelerator and mixed again. After a homogeneous mixture was obtained, the mixture was poured into the Teflon mold placed on a smooth surface. The Teflon mold was kept at 85°C for 10 minutes and the hardened samples were removed from the mold. These procedures were applied in the same way for the other ratios.

2.3. Characterization

The tensile test of the samples prepared according to the ISO 527 standard was carried out in the Zwick Z010 universal tensile device with a tensile speed of 5 mm/min. The 3-point bending test was also performed on the Zwick Z010 device. The impact strength of the unnotched samples prepared according to the ISO 180 standard was tested using a 5.4 J Izod hammer in the Zwick B5113.30 impact device. Hardness measurements were made on the Zwick Shore D instrument. The abrasion test of the samples was carried out in a Pin On Disc Tester according to ASTM G-99 standard under 250 g weight load at 72 rpm. Weight measurement was carried out by taking a measurement every 100 meters (100 m = 398 cycles), for a total of 500 m (total 1990 cycles). In the wear test, an abrasive disc with a hardness of 63 HRC and a surface roughness of 0.830 µm was used. The weight loss was determined by measuring with a Precision Balance with a precision of 10^{-4}. SEM examination of the fractured surfaces was performed with the JEOL JSM-5910 LV device.

In the tests performed, 5 samples were prepared for each group, the average of the resulting values was taken and the tests were carried out at room temperature.

3. Results and Discussion

In this study, Al_2O_3 particles of 1-3 micron dimensions were used as a reinforcement in vinyl ester matrix composites and their mechanical properties were investigated. The test results of the samples are given in the figures below.

Tensile test results are given in Figure 1. As can be seen in Figure 1, the tensile strength increased with an increase in the amount of aluminum oxide added to the vinyl ester up to 1.5%, after which a decrease was observed in the tensile strength. It was seen that the maximum % elongation was observed in the sample containing 1.5% Al_2O_3 as in the tensile strength (Figure 2). The non-homogeneous distribution of Al_2O_3 particles in the composite structure and the formation of agglomeration caused the expectation of higher tensile strength to not be met. It is seen that the increase in strength in microparticle reinforcement is not higher and the decrease in strength by 2% is due to inhomogeneous distribution. It is seen that this is due to the stress concentration that occurs by causing agglomeration.

In some studies, it is seen that silica nano particles are added to epoxy resin for the purpose of toughening [8], and in another study, boron nitride is added to vinyl ester resin for the same purpose [17]. In these studies, it is stated that silica and boron nitride increase toughness. In this study, adding aluminum oxide to vinyl ester resin had the same effect on increasing toughness. In the study of Latief F.H.. et al. [18], it is seen that the tensile strength increases up to 5% with the addition of Al_2O_3 into the polyester resin, and after this rate, the tensile strength decreases due to agglomeration. The same effect was observed in our study.

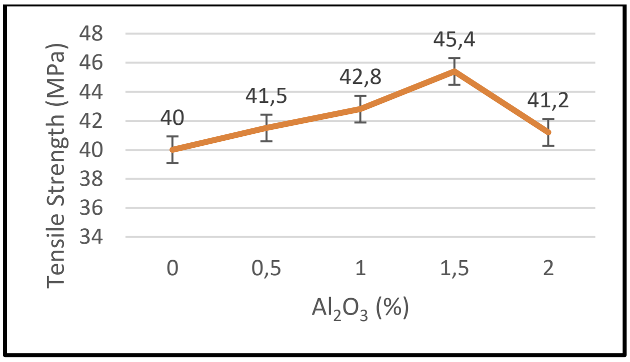

Figure 1. The change in tensile strength depending on the amount of Al_2O_3

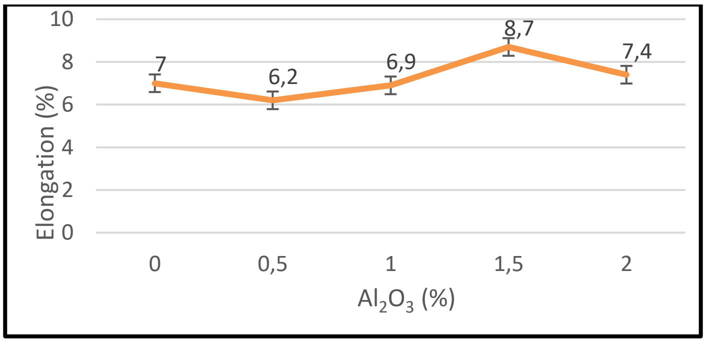

Figure 2. The change in % elongation depending on the amount of Al_2O_3

Figure 3 shows the three-point bending test results depending on the addition of aluminum oxide. As the aluminum oxide addition increased, the bending strength decreased. As expected, the highest values were obtained in the pure vinyl ester sample. Depending on the increase in the amount of Al_2O_3 particles, the hardness increased and this caused the composite material to have a brittle structure. The increase in hardness and the formation of agglomeration caused a decrease in bending strength.

In the study of İskender et al. [13], it is seen that the bending strength decreases as the amount of Al_2O_3 added to the epoxy resin increases. The same effect was seen in this study.

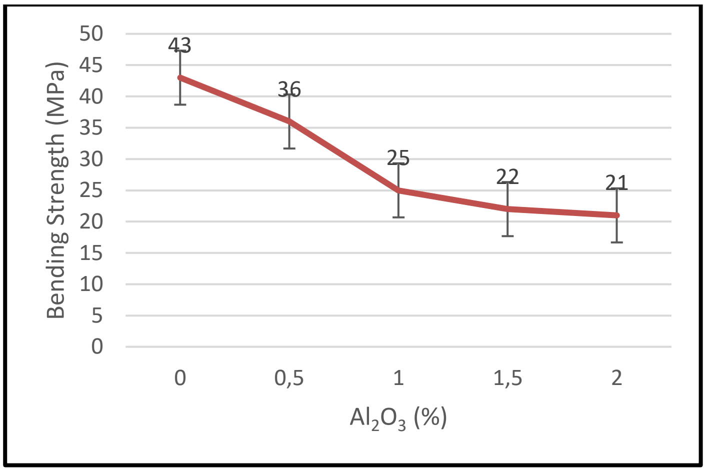

Figure 3. The change in the three point bending strength of the vinyl ester/Al_2O_3 composites

In Figure 4, the change in Izod impact strength depending on the amount of aluminum oxide is given. In this study, the maximum impact strength was obtained in the 1.5% Al_2O_3 reinforced sample. After this ratio, the impact strength decreased. In the impact test, it was observed that the vinyl ester matrix fracture in the brittle fracture mode without undergoing significant plastic deformation. As it is known, as the particle size decreases, the surface area increases and the adhesion between the matrix and the reinforcement phase is better. On the other hand, as the amount of particles increases, the probability of formation of agglomeration also increases. In this study, the impact strength increased with the addition of Al_2O_3, which has a small particle size of 3 microns. The maximum impact strength was observed at a rate of 1.5%.

Aynalem G.F. and Sirahbizu B. [19] added Al_2O_3 into the linen/unsaturated polyester composite structure in their study. They stated in their studies that the impact strength increased with the increase of the amount of Al_2O_3. In our study, the impact resistance increased with the increase in the amount of Al_2O_3 added to the polyester resin.

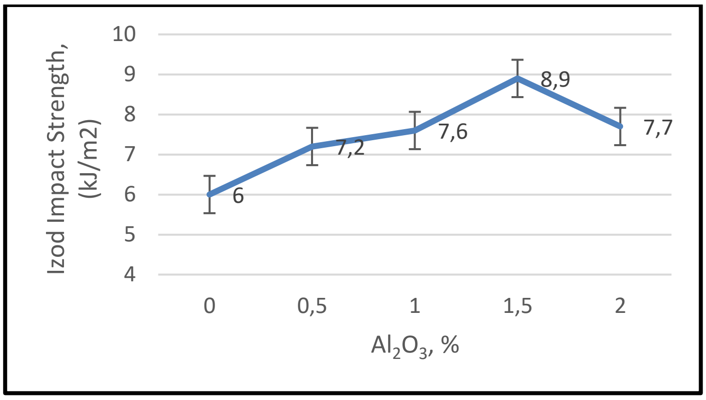

Figure 4. The change in the izod impact strength of the vinyl ester/Al_2O_3 composite

In Figure 5, hardness values are given depending on the amount of aluminum oxide. Although the hardness value increases with the increase of Al_2O_3 ratio, this increase is not a serious increase. However, the slight increase in hardness caused to have a brittle structure of the composite materials.

In the study of Latief F.H. et al. [18], it was observed that the addition of Al_2O_3 increased the hardness of the polyester resin. Al_2O_3 showed the same effect in vinyl ester resin.

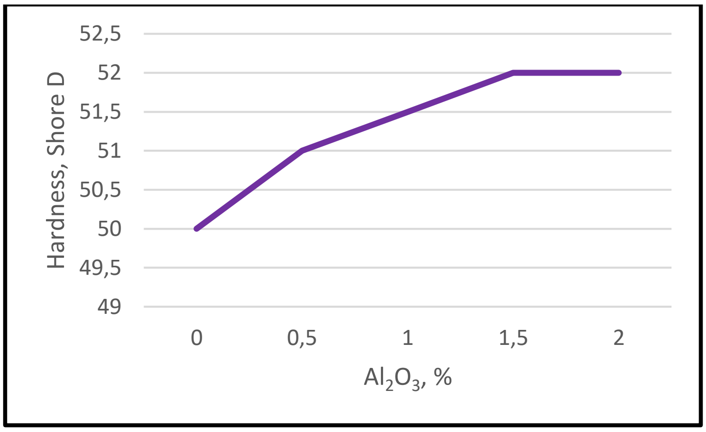

Figure 5. Hardness values depending on the amount of Al_2O_3

The weight losses obtained as a result of the wear test are given in Figure 6. Weight loss decreased depending on the increase in the wearing distance and the % aluminum oxide ratio. While it is seen that the most wear is in pure vinyl ester resin (0%), it is seen that the amount of wear decreases with the addition of aluminum oxide, that is, the wear resistance increases. The wear resistance of the 1.5% and 2% Al_2O_3 reinforced composite materials is higher than the other samples. Due to the good adhesion between vinyl ester and Al_2O_3, the wear resistance increased as the amount of Al_2O_3 increased.

In the study of Solmaz et al. [14], the addition of Al_2O_3 to the polyester resin increased the abrasion resistance. In our study, the addition of Al_2O_3 showed the same effect in vinyl ester resin.

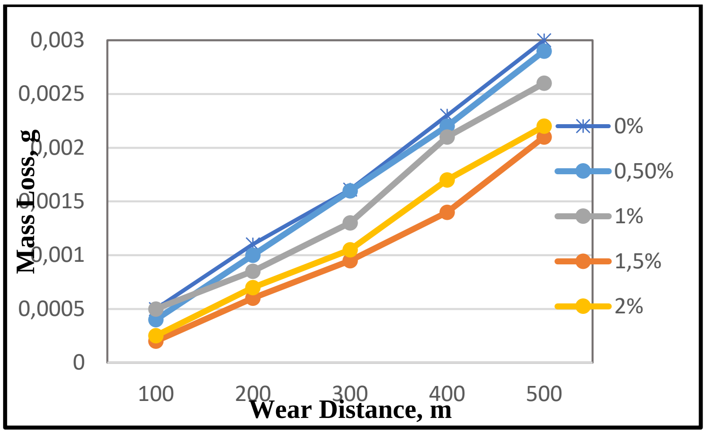

Figure 6. Wear test results of the vinyl ester/Al_2O_3 composites

A fractured surface view of vinyl ester composite with 2% Al_2O_3 added is given in Figure 7. When the distribution of the particles in the structure is examined, it is seen that particle distribution is observed in some regions, while agglomeration occurs in some regions. In other words, a heterogeneous distribution was obtained. Despite adding MEG and mixing in the ultrasonic homogenizer to prevent agglomeration, agglomeration still occurred in some areas, which is due to the difficulty of homogeneous distribution as the amount of Al_2O_3 increases. This explains the reason for the decrease in the mechanical properties of 2% Al_2O_3 reinforced composites. Figure 8 shows the result of the EDS test and the Al and O peaks can be seen in the graph.

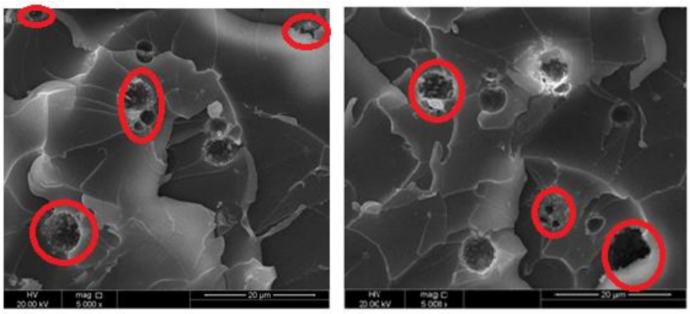

Figure 7. SEM image of 2% Al_2O_3 reinforced vinyl ester composite

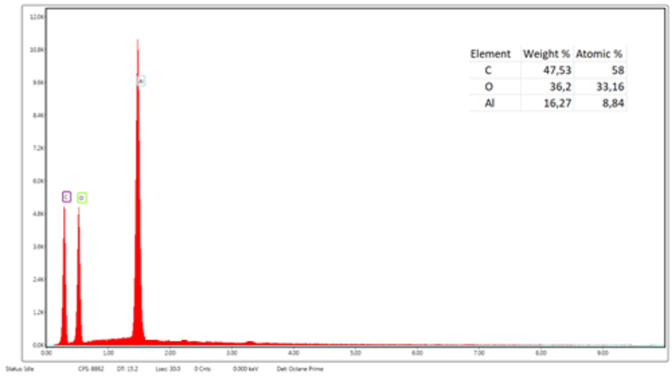

Figure 8. EDS test of reinforced vinyl ester composite

4. Conclusions and Recommendations

Al_2O_3 particles (1-3 micron) were used as a reinforcement in vinyl ester matrix composites and their mechanical properties were investigated. The tensile strength, % elongation, hardness and impact strength properties of the composite material increased with the addition of aluminum oxide. According to these mechanical properties mentioned here, the best result was seen at a rate of 1.5%. However, the flexural strength decreased as the Al_2O_3 ratio increased. As for the abrasion properties, the mass loss was decreased as the content of Al_2O_3 increased. In the abrasion test, the ratio of 1.5% Al_2O_3 gave the best abrasion resistance result.

In this study, it was understood that the addition of Al_2O_3 had a significant effect and improvement on most of the mechanical properties of vinyl ester composites, but this effect reached the optimum effect at the level of 1.5%. The addition of reinforcement at high rates in micron-sized particles causes a weakness in the adhesion force between the matrix and the reinforcement, while on the other hand, it also causes agglomeration problems. Therefore, a decrease in the mechanical properties of vinyl ester composite occurred after 1.5% Al_2O_3 ratio. The results of this study are also consistent with the results of other studies in this field.

Al_2O_3 reinforced vinyl ester composite can be used in areas where mechanical properties other than bending strength are important. In addition, this method is recommended for producing parts with small dimensions and recessed protruding geometries, which are reinforced in the independent direction. The experimental results support that the successful addition of Al_2O_3 particles reinforced vinyl ester composites is possible to fabrication.

In terms of homogeneous distribution, non-agglomeration, better adhesion of matrix and reinforcement, the use of particles such as Al_2O_3 in nano size will give better results in terms of improvement of mechanical properties.

5. Acknowledge

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