Effect of Milling Time on Alumina-Titania-Carbon Nanotube by Powder Metallurgy Method

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Abstract. The Recent work discovers the evaluation on alumina-titania-carbon nanotube (Al₂O₃–TiO₂–CNT) composite prepared by powder metallurgy method. The properties of Al₂O₃–TiO₂–CNT composite have high hardness, chemical and thermal resistance, wear and corrosion resistance but lacking to low fracture toughness which lead to brittle properties. Hence, CNT was added to the system due to attractive mechanical properties, low density and high fracture toughness. Elemental powders of Al₂O₃–TiO₂–CNT were milling in low energy ball milling using different milling time at 15, 30, 45 and 60 hours. The peak XRD of TiO₂ and CNT become diminished with increasing milling time. The powder that milled at 60 hour possible to has lowest crystallite size and highest internal strain due to finer and homogenous particle size deform during milling.

Keywords- Alumina, titania, carbon nanotube, powder metallurgy, compaction and milling

1. Introduction

Composites will be defined as one or more discontinuous phases embedded in a continuous phase on macro scale that can improve the properties of material [1]. The discontinuous phase are called reinforcement is usually harder and stronger than the continuous phase that are called as matrix. Composite will be divided into three classifications which are metal matrix composite (MMC), polymer matrix composite (PMC) and ceramic matrix composite (CMC).

CMC is material that promises as potential candidates in view of excellent physical and mechanical properties. This is due to their properties like high temperature stability, chemical inertness, strength and corrosion resistance that appropriate usage in industry [2-3]. In previous years, many researchers have been carried out to produce CMC in advance material that used in various industries such as electrical, medical application, aerospace and automotive part [4].

Alumina (Al₂O₃) ceramic material is mostly used as matrix for CMC application because of their superb properties such as high hardness, high compressive strength (0.5-5.0 wt.%) and good chemical resistance. This was proved according to many of CMC that applied of Al₂O₃ based ceramic as matrix material. For example, Al₂O₃ based silver (Al₂O₃–Ag) that improve fracture toughness in range 3.2–MPa·m¹/₂ and Al₂O₃ based silicon carbide (Al₂O₃–SiC) [5].

Based on research that was reported [6], alumina–titania (Al₂O₃–TiO₂) is one of CMC that already fascinating study by others due to good ceramic material for many applications such as machinery, textile and printing industries. Al₂O₃–TiO₂ also has tremendous properties due to their high hardness, excellent wear corrosion, chemical and thermal resistance. However, Al₂O₃–TiO₂ composite have
limitation leading to poorer fracture toughness and high porosity because of not fully homogeneous of the grain size.

In order to improve the properties of $\text{Al}_2\text{O}_3$-$\text{TiO}_2$ composite, carbon nanotube (CNT) will be added to become $\text{Al}_2\text{O}_3$-$\text{TiO}_2$ systems prepared by powder metallurgy (PM) technique. CNT play an important role in order to improve the $\text{Al}_2\text{O}_3$-$\text{TiO}_2$ composite because of extent excellent properties such as high tensile strength in range of 10–100 times greater than steel [7], low density ($\sim 2.1 \text{ g/cm}^3$), light weight, promising stiffness, Young’s modulus (973 GPa) and high strength (up to ~100 GPa) [8-9].

2. Methodology

The material used in this experiment were $\text{Al}_2\text{O}_3$ (> 99.9% purity, average particle size > 20 µm), $\text{TiO}_2$ (> 99.5% purity, average particle size > 21 nm) and CNT (99.9% purity, average particle size > 20 µm). The composition was used in this study to prepare $\text{Al}_2\text{O}_3$–$\text{TiO}_2$–CNT composite and has been purchased from Sigma Aldrich Company.

3. Results and Discussion

The characteristic of pure powders were analysed using XRD. Figure 1 shows the XRD pattern of $\text{Al}_2\text{O}_3$, $\text{TiO}_2$ and CNT. Based on XRD result, the pure powders were analysed to have crystalline structure. The $\text{Al}_2\text{O}_3$ powder was observed that have four strong peaks at 25.62°, 35.21°, 43.43° and 57.61°. While, the $\text{TiO}_2$ analysis shows the $\text{TiO}_2$ powder also have three sharp peaks at 25.31°, 36.92° and 48.04°. However, the other peak appeared show no strong because of short and broadening.

Figure 1: XRD patterns of (a) $\text{Al}_2\text{O}_3$, (b) $\text{TiO}_2$ and (c) CNT powder

The $\text{Al}_2\text{O}_3$ revealed as corundum phase while $\text{TiO}_2$ showed well defined diffraction peaks corresponding to anatase phase, that form stable rather than rutile and brookite. This similar with the previous research [7]. For both of $\text{Al}_2\text{O}_3$ and $\text{TiO}_2$ peak exhibit the strongest peak are at 35.03° and 35.15° that also present same to previous study that revealed in Figure 2.10. In Figure 4.1 the crystalline peak of CNT powder, highest intensity is obtained at position 24.66° and short peak at 44.67. Both crystalline peak of CNT powder was show not strong peak, this is due to high solid solubility of CNT and peak found in amorphous structure [10].

Figure 2 shows the result of XRD pattern of $\text{Al}_2\text{O}_3$–$\text{TiO}_2$–CNT powder mixture at different milling time (15, 30, 45 and 60 h) with 10 mm alumina ball. $\text{Al}_2\text{O}_3$ and $\text{TiO}_2$ phases are observed having
The crystalline structure while CNT phase has very weak peak obtained after milled. The crystalline phases were obtained with different milling time provide no sign of amorphous phases have seen in diffraction peak for all milling time. The first crystalline peak of Al$_2$O$_3$, TiO$_2$ and CNT indicate was overlapped each other, due to their peak are seen much closed at 15 and 30 h of milling time. The peak crystallite structure of Al$_2$O$_3$ was seen at 25.57° for both 15 h and 30 h. The peak TiO$_2$ phase was presented at 25.30° for 15 h and small shifted to the left at 25.48° and no difference of 2θ for CNT peak at 25.63° for both 15 h and 30 h.

Based on Figure 2, the four strong peaks of Al$_2$O$_3$ has appeared at hkl ([101], ([114]), ([213]), and ([216]) become shorten and diminished with increasing milling time. Besides that, the peak of Al$_2$O$_3$ also reduce size of particle and become more wider according to the grain size become refining with increasing of milling time [11] - [12]. Peak of TiO$_2$ also appeared at hkl ([010], (004), and ([005]) and slightly diminished due to small size with increasing the milling time. Apart from that, TiO$_2$ phase was easy to diffuse into matrix phase of Al$_2$O$_3$. CNT has shortened peak and not observed at all milling times, presumably as a result of its faster diffusion into the Al$_2$O$_3$ matrix than TiO$_2$. That is, solid solubility for CNT in Al$_2$O$_3$ is far higher than that of TiO$_2$, and can be later diffused into a solid Al$_2$O$_3$ solution. Moreover, the evidence the changes of Al$_2$O$_3$ lattice can be described by shifted Al$_2$O$_3$ peaks to the left with prolonged milling time.

Figure 2. XRD patterns of milled powder at (a) 15 (b) 30 (c) 45 and (d) 60 h

Brittle phase powder basically dissolves into ductile matrix phase during milling. Two methods was used to describe the solubility of powder, there are peak disappearance and peak shift. In this experiment, the methods were used to discuss the solubility of Al$_2$O$_3$, TiO$_2$, and CNT in Al$_2$O$_3$ again milling time. The formation of second element into matrix phase of Al$_2$O$_3$ was discovered by peak disappearance in XRD pattern. The peak shifts either to the right or left of $2\theta$ angle axis is also another method to determine dissolution of second phase into matrix lattice.

In this experiment, no visible peak shifting is observed at early milling time. This due to low energy ball milling was used at only 200 rpm and shorten milling time that not enough to form new phase of solution. This can explain by limitation amount of Al$_2$O$_3$ solution that not contributes to the change of Al$_2$O$_3$ lattice. There was no transformation of TiO$_2$ and CNT into Al$_2$O$_3$ phase during the milling process. Thus, it can discovered that peaks of Al$_2$O$_3$ and TiO$_2$ are show broadened.
Besides, the wider peak occurs due to internal stress accumulation induced by formation and the presence of tiny particle of second phase that introduce defect. The crystallite size and internal strain of powder mill not easy to change by milling time 15, 30, 45 and 60 h due to little shift peak to left was formed. Based on result, the 60 h of milling time provide strong formation of second phase of $\text{Al}_2\text{O}_3\text{–TiO}_2$–CNT powder.

4. Conclusion
As a conclusion, based on XRD pattern no visible new phase was formed when increasing milling time with only the changes of lattice parameter and crystallite size. The lowest crystallite size of and highest internal strain $\text{Al}_2\text{O}_3\text{–TiO}_2$–CNT are at 60 h.

5. References
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