

Fabrication and property evaluation of WO₃ particles dispersed Al-based composite material

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Abstract. Advantage of composite material is to take the outstanding mechanical and functional properties of fibers and powders. However, it is not easy to disperse fine particles homogeneously in metallic matrix. 3-dimensional penetration casting (3DPC) method enables to disperse particles in the matrix homogeneously without segregation. The present study puts its focus on WO₃ particle. Several reports are available about photocatalytic property of WO₃ in the visible ray region. Photocatalytic is one of the most promising matter that can be used for resolving environment pollution because photocatalytic cause the oxidation reaction that change carbon contained in organic compound (for example acetaldehyde, toluene) into CO₂ by light. The aim of this work is to establish the fabrication method using 3DPC, to evaluate the mechanical, photocatalytic properties, and to observe the microstructure using scanning electron microscopy (SEM), transmission electron microscopy (TEM).

1 Introduction

Metal matrix composites (MMCs) have wide range of applications in industrial fields such as automotive and aerospace industry because MMCs have the advantage to take the outstanding mechanical properties and functional properties of fibers and particles. There are some fabrication methods of particle reinforced MMCs, for example powder metallurgy [1], stir casting [2], squeeze casting and spray forming [3]. It is known that fabricating MMCs by casting method is difficult to obtain without particle aggregation, a high-volume fraction of particle, fine particles below 5 μ m [4]. However, three-dimensional penetration casting method (3DPC) enables to resolve the problems as mentioned above. In our previous work, we fabricated the MMC using photocatalysis particles (TiO₂) and pure Al. Photocatalysis has been attracted and a lot of researches have done because photocatalysis is expected to resolve this environment pollution problems due to ability of decomposition for organic pollution by light irradiation. TiO₂ is the most widely used as photocatalysis because of its application for environmental purification [5,6]. However, the band gap of TiO₂ about 3.2eV limits its application only in ultraviolet region [7]. To response in visible ray region, we focused on WO₃. WO₃ has a wider response in the solar spectrum because of its lower band gap of 2.8eV [8] compared to TiO₂, and it is expected as a visible light active photocatalyst [9].

In this research, Al-based WO₃ particles dispersed composite material was fabricated using 3DPC process

and micro-structure observation was performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Mechanical and functional properties were estimated by means of hardness measurement and photocatalytic property test.

2 Experimental procedure

WO₃ powders (A.L.M.T Corp) with average size of 5 μ m~50 μ m, and 99.99% purity aluminium were employed. High WO₃ volume fraction of Al-based composite material was fabricated by using 3-dimensional penetration casting (3DPC) process. WO₃ powders were packed and put on the steel mould. Molten Al heated up to 1053K was poured into steel mould, and pressed from a top. After cooling, the composite material was removed from the steel mould. Volume fraction of WO₃/Al composite material was measured by Archimedes method. The microstructure observation was conducted by SEM, (Hitachi S3500H) equipped with an energy dispersive spectroscopy (EDS). To investigate the reaction products at interface between WO₃ and Al with high magnification, TEM (JEOL 4010T operated at 400KV) was used. TEM sample was prepared by focus ion beam (FIB). The mechanical property was evaluated by Vickers micro-hardness test. Vickers micro-hardness test was carried out on polished sample using Mitutoyo HM-101 with a load of 100g and holding time of 15s. Photocatalytic property was evaluated photocatalytic property evaluation checker PCC-1 (ULVAC PIKO Co. Ltd.) that measured the

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change amount of ultraviolet radiation absorbance of methylene blue aqueous solution on the surface of composite material with ultraviolet irradiation. 20×10^{-3} mol/L methylene blue aqueous solution was prepared by dissolving methylene blue trihydrate in distilled water. Samples were dipped in methylene blue aqueous solution for 1h and drying for 10h. To improve the photocatalytic property by increasing WO_3 ratio at surface area, the electro-polishing was performed for WO_3/Al composite material at 253K with a solution of 10% perchloric acid and 90% ethanol.

3.Result and discussion

3.1. Microstructure observation

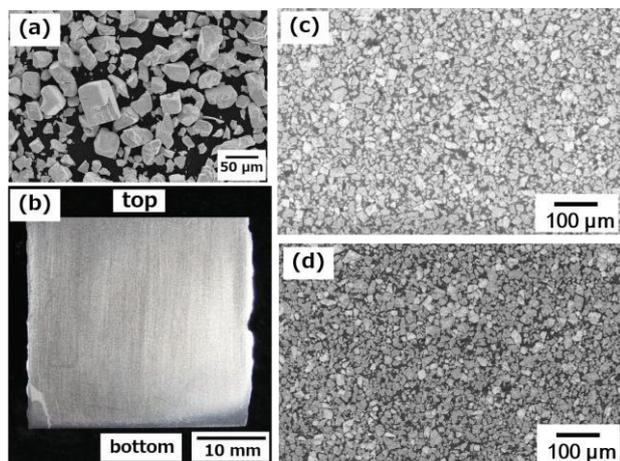


Figure 1 (a) SEM image of powder, (b) cross-sectional image of WO_3/Al composite material and SEM image of (c) middle area and (d) bottom area of WO_3/Al composite material.

Figure 1 (b) shows the cross-sectional image of WO_3/Al composite material and SEM images of (c) middle area and (d) bottom area. The dark and white contrast regions correspond to Al matrix and WO_3 particles, respectively. It is noted that there are no clack, sink and unpenetrated part of Al. WO_3 particles were dispersed homogeneously in Al matrix after casting. It is measured that the volume fraction of WO_3 particles was 66% by Archimedes' principle. WO_3 particles size of composite material maintain the initial particle size, Fig.1(c), (d). This result shows that WO_3 particles were dispersed into Al matrix without aggregation by 3DPC process.

3.2 Mechanical property

The results of Vickers micro-hardness measure for pure Al and WO_3/Al composite material are 23HV and 101HV, respectively. Hardness level of WO_3/Al composite material is about 4 times higher than pure Al. Figure 2 shows the indentations after hardness test of WO_3 area and Al area. The hardness of WO_3 and Al were 163HV and 48HV in Fig.2, respectively. The hardness level of WO_3 is much lower than that of WO_3 sintered sheet [10] and it was caused by aggregation of WO_3 particles. Above $50 \mu m$ particles was observed in Fig.1 (a) because of aggregation, and it results in reduction the hardness of

WO_3/Al composite material. It should be noted that the hardness of Al area was twice higher than pure Al. That was explained by results of an EDS analysis. Mainly WO_3 area and Al area were separated clearly, however fine WO_3 particles were detected in Al area, which are marked by white arrows in Fig.3 and it is possible to enhance hardness of Al matrix.

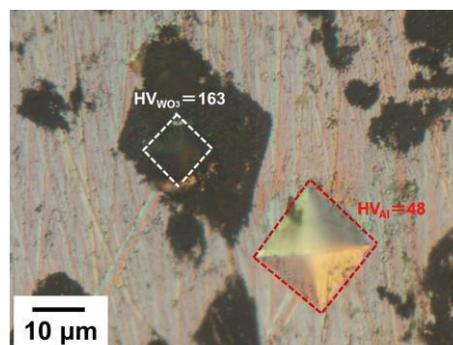


Figure 2. Indentations of Vickers micro-hardness test for WO_3 and Al area.

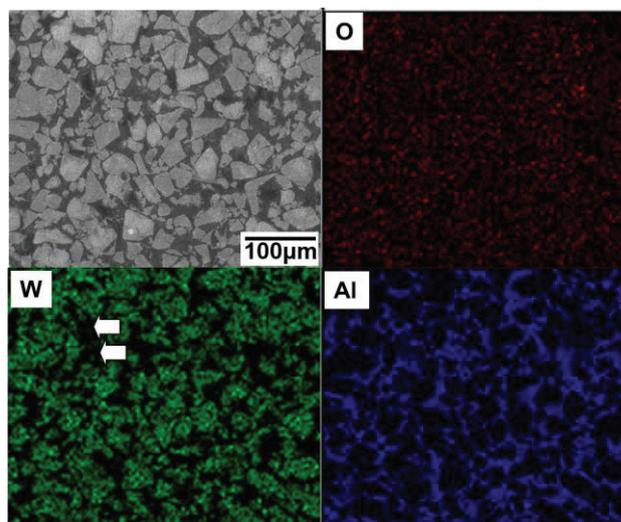


Figure 3. EDS analysis for WO_3/Al composite material and white arrows exhibit fine WO_3 particles detected in Al area.

3.3 Photocatalytic property

Figure 4 shows the result of photocatalytic property evaluation. The lower ΔABS (the varied amount of absorbance) is, the higher photocatalytic property exhibits. Lowering of ΔABS was also observed in WO_3/Al composite material. However, photocatalytic property level of WO_3/Al composite material was inferior WO_3 . It can be considered that the effect of reaction products between WO_3 and Al that were caused during casting process. To clarify the existence of reaction products at interface between WO_3 and Al, TEM observation and an EDS analysis were conducted to investigate the reaction products at interface between WO_3 and Al. Figure 5 shows the TEM image of interface between WO_3 and Al and the results of EDS analysis that was carried out on WO_3 and Al area close to interface. The region dark and white contrasts correspond to WO_3

matrix and Al particles, respectively. As Figure 6 indicated, the interface between WO_3 and Al was relatively clear. In EDS analysis, W, O and Cu peaks were arisen in area 1 (WO_3 area) and Al, O and Cu peaks were detected area 2 (Al area) in Fig.6 both Cu peaks came from Cu mesh. Based on these results, it is considered that there were no reaction products between Al and WO_3 .

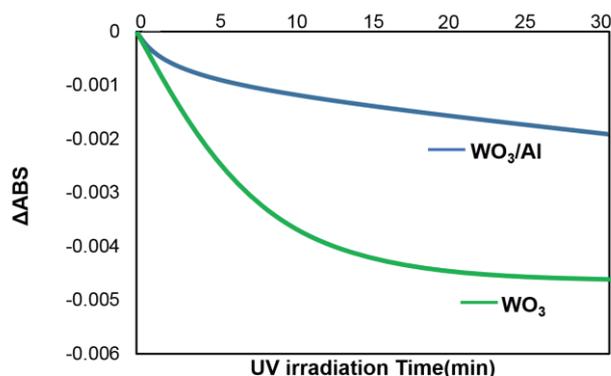


Figure 4. Photocatalytic property evaluation for WO_3 and WO_3/Al composite material.

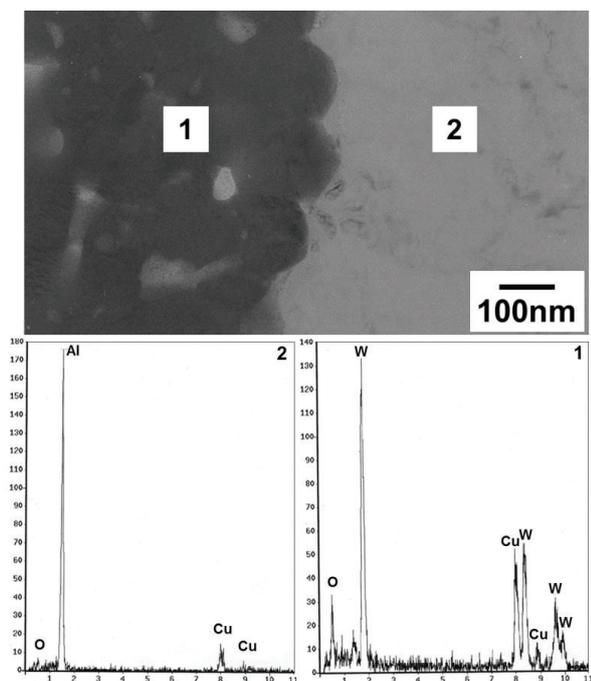


Figure 5. TEM observation at interface between WO_3 and Al and EDS analysis in WO_3 area and Al area close to interface.

To improve the photocatalytic property level, the electro-polishing was conducted. It enables to cause preference etching of Al and to expose more WO_3 particles on surface. Figure 7 shows microstructure changing by polishing method from (a) mechanical polishing to (b) electro-polishing. Green contrast and red contrast correspond to Al area and WO_3 area, respectively. WO_3 ratio on the surface in mechanical polishing and electro-polishing were 66% and 91%, respectively. In comparison with mechanical polishing, WO_3 ratio on the surface in electro-polishing indicated about 30% higher. Photocatalytic property evaluation of WO_3/Al composite

material after electro-polishing appears in Figure 8. ΔABS of electro-polished sample became better than mechanical polishing. It is interpreted from this result that electro-polishing enhances photocatalytic property of WO_3/Al composite material by increasing WO_3 ratio at surface area.

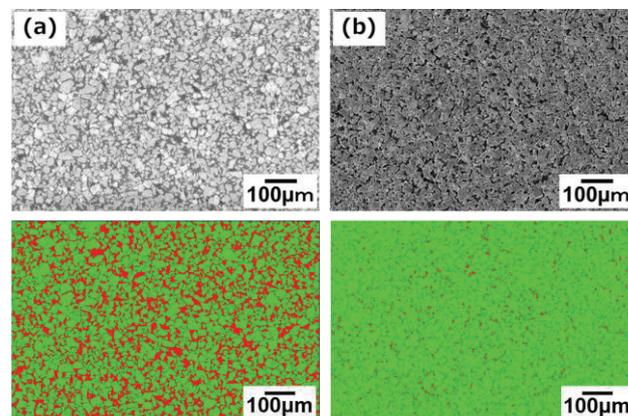


Figure 6. SEM images of WO_3/Al composite material changing polishing method from (a) mechanical polishing to (b) electro-polishing. Green and red contrasts correspond to WO_3 and Al area.

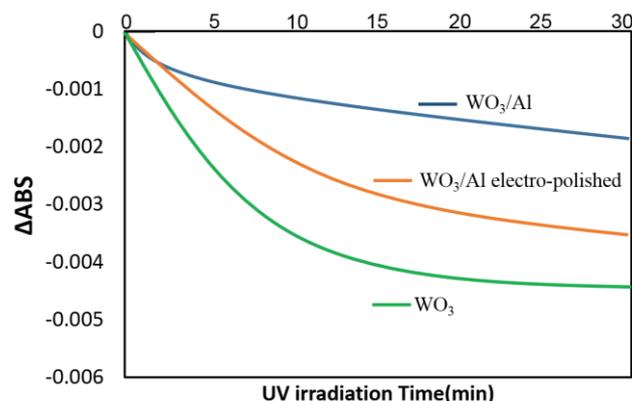


Figure 7. Photocatalytic property evaluation for WO_3 , WO_3/Al composite material and after electro-polishing.

4 Conclusions

- (1) WO_3 particles dispersed Al-based composite material was successfully fabricated by 3DPC process. In results of micro-structure observation, it was found that there is no unpenetrated part of Al, and WO_3 particles were dispersed into Al matrix homogeneously. The difference of distribution of WO_3 particles on places was not confirmed and it results in distribution of WO_3 particles without segregation.
- (2) The hardness of composite material shows four times higher than pure Al. The hardness of Al area in composite material was twice higher than pure Al because fine WO_3 particles penetrated Al matrix. Those particles enhanced hardness level of Al matrix.
- (3) The photocatalytic property was confirmed in composite material. By electro-polishing, WO_3 particles were exposed from surface because of preference etching of Al. electro-polished sample

showed better photocatalytic property than mechanical polishing.

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