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## 学位論文の概要及び要旨

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題 目 Processing and Characterization of In-situ Synthesized and Hot-extruded Aluminum Matrix Composites Reinforced with Al<sub>2</sub>O<sub>3</sub>-TiB<sub>2</sub>-TiC Ceramic Particles (In-situ 合成と熱間押出しにより合成したセラミック粒子強化アルミニウム基複合材料のプロセッシングと評価に関する研究)

### 学位論文の概要及び要旨

Aluminum-matrix composites (AMCs) have found widespread applications in various fields including aerospace, automobile and maritime industries due to their high specific strength and modulus, good wear and heat resistance, and low coefficient of thermal expansion. As a result, there is need to further enhance these properties to meet the increasing demand. However, conventional processing techniques like stir casting and powder metallurgy result in AMCs with large particulates in the order of microns to tens of microns which are usually nonuniformly distributed within the Al matrix. Further, most common AMCs are reinforced with single particulates rather than hybrid reinforcements.

In-situ synthesis of hybrid particle reinforcements followed by hot-extrusion densification process emerges as the best technique of producing AMCs with fine particles which are uniformly distributed within the Al matrix leading to improved microstructure for better mechanical properties resulting from strong bonding and wettability between the matrix and reinforcement phases occasioned by clean and clear interfaces. In this study, AMCs reinforced with submicron ceramic particles of Al<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub> and TiC were in-situ synthesized by reactive sintering of Al/AA6061, TiO<sub>2</sub> and B<sub>4</sub>C powder mixtures and further densified by a hot-extrusion process. The reaction mechanisms for the formation of reinforcing particles, extrusion behavior, microstructure, hardness, wear behavior, fatigue strength and tensile properties (room and high temperature) were investigated.

To study the reaction mechanisms and reaction path in the Al-TiO<sub>2</sub>-B<sub>4</sub>C system, the starting powders were weighed stoichiometrically to produce AMCs with target reinforcing particulates at 15 vol%, 20 vol% and 25 vol%. The reactions of TiO<sub>2</sub> and B<sub>4</sub>C with molten Al were a stepwise process, and there were many intermediate phases including oxygen deficient titanium oxides (Ti<sub>3</sub>O<sub>5</sub>,

Ti<sub>2</sub>O<sub>3</sub> and TiO), Al<sub>4</sub>C<sub>3</sub>, AlB<sub>2</sub> and Al<sub>3</sub>Ti, before the expected reinforcing particles of Al<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub> and TiC were formed. Submicron particulates with an average particle size of 0.24 μm were uniformly distributed in the matrix.

The porous reactive-sintered AMCs with 85 - 92% relative density (RD) were successfully densified through hot extrusion in a temperature range of 480°C-550°C to a maximum of 99.8%. Furthermore, the microstructure of the resulting AMCs was characterized by fine reinforcing ceramic phases with an average particle size of 0.24 μm, which were homogeneously distributed in Al matrix. The ceramic particulate content and extrusion temperature have been proved to have small influences on the average particle sizes of the reinforcing phases, but a substantial decrease in Al grain size with increase in ceramic particle contents and decrease in extrusion temperature was observed.

The effects of extrusion temperature and ceramic particle contents on mechanical properties including micro-hardness, fatigue strength and wear resistance were also studied. As the ceramic particle contents increased and extrusion temperature decreased, RD, micro-hardness, wear resistance and fatigue strength improved. Maximum micro-hardness of 145.6 HV was achieved at 25 vol% ceramic particle content and extrusion temperature of 520°C representing an improvement of 219.1% from the hardness values of pure aluminum (45.6 HV) prepared by powder metallurgy process and extruded at the same temperature.

The tensile strength and yield strength improved with increase in ceramic particle content and decrease in extrusion temperature. The 25 vol% AMC extruded at 520 °C AMC had the highest average tensile strength of 425.0 MPa which was 9.6% higher for the AMCs with the same ceramic particle content but extruded at a higher temperature of 550 °C (388 MPa). The improved strength was caused by the strengthening mechanisms especially the Orowan and CTE mismatch strengthening which accounted for about 15% and 64% of the estimated theoretical yield strengths respectively. The fracture surface of pure Al extruded at 550 °C exhibited ductile fracture morphology consisting of large dimples and voids. The fracture surfaces of the AMCs were characterized by smaller and shallower equiaxed dimples with tear ridges. Further, high temperature tensile properties were conducted at 300 °C and 370 °C. Even though precipitation-hardened aluminum alloys AA6061-T6 and AA2024-T3 had 24.2% and 93.5% better tensile strength at 25 °C, 15 vol% AMCs extruded at 550 °C gave a 66.7% and 44.5% better tensile strength at 300 °C and 370 °C respectively.

To further enhance the properties of the AMCs, Al matrix was alloyed to the AA6061 standard and the reaction mechanism with TiO<sub>2</sub> and B<sub>4</sub>C studied. The formation of the target reinforcing ceramic particles were completed at 1400 °C without intermediate compounds. Uniformly distributed fine ceramic particles were observed in the AA6061 matrix which appeared larger than those formed in pure Al matrix. The reactive sintered AMCs had several pores (RD ~ 83%) which were positively reduced by hot-extrusion secondary processing to produce composites with improved relative density

(about 100%) and microstructures. The microstructure revealed that the ceramic particles of  $\text{Al}_2\text{O}_3$ ,  $\text{TiB}_2$  and  $\text{TiC}$  were uniformly distributed within the alloy matrix. The average particle sizes of 1.03  $\mu\text{m}$  was realized.

The dense AA6061 matrix composites exhibited improved micro-hardness which increased with increase in ceramic particle contents and extrusion temperature. A maximum hardness of 110.8 HV was realized at an extrusion temperature of 520 °C. The UTS and YS of the AA6061 matrix composites increased with increase in ceramic particle content while ductility decreased. There was little effect of extrusion temperature on mechanical properties. The highest tensile strength of 266.5 MPa was achieved at 20 vol% ceramic particle content and extrusion temperature of 550 °C. The micro-hardness and wear resistance of the AA6061 matrix composites greatly improved with the increase in ceramic particle content.

The AA6061 matrix composites were heat-treated to the T-6 standard and the effect of heat-treatment on microstructure, strength, hardness and wear properties investigated. There was little effect of heat-treatment on the microstructure of the AA6061 matrix composites, however, the presence of ceramic particles leads to further grain refinement. The heat-treated AA6061 matrix composites demonstrated higher hardness than the non-heat-treated alloy composites. The peak-ageing time for the AMCs corresponding to peak-hardness increased with increase in the ceramic particle content. The peak-ageing time for 0 vol%, 15 vol% and 20 vol% AMCs were 4 h, 8 h and 12 h respectively. The UTS and YS improved with increase in ceramic particle contents for the non-heat-treated AMCs. The response to heat treatment was larger for unreinforced AA6061 matrix composites which improved to 322 MPa. The heat treated 15 vol% AMC demonstrated a lower UTS and YS than the heat treated and AA6061 Al alloy with 0 vol% ceramic particle content.