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# Friction stir welding of multi-walled carbon nanotubes reinforced Al matrix composites

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ABSTRACT

In this study, we prepared composites between Al and multi-walled carbon nanotubes (MWCNTs) using the friction stir welding (FSW) method and investigated their microstructural evolution and mechanical properties by controlling the amount of MWCNT (1 and 3 vol%) and the plunging load (400 and 600 kg) used. After the FSW process, MWCNT showed slight coarsening of grain with random shear textures and accumulated dislocations; the differences obtained by varying the plunging load and MWCNT amount were insignificant. The friction-stir welded composite obtained using 3 vol% MWCNT and a plunging load of 600 kg possessed two-times better balance between strength and ductility than the base metal, due to the presence of both MWCNTs and the partially decomposed $Al_4C_3$ in the composite.

## 1. Introduction

Composites containing an Al matrix reinforced by multi-walled carbon nanotubes (MWCNTs) have attracted the attention of aerospace and automotive industries because of their high strength-to-weight ratios owing to the efficient load transfer of MWCNTs in the matrix [1–6]. Many researchers have extensively studied the optimization of the mechanical properties of Al/MWCNT composites by tuning the amount of MWCNT using a powder metallurgy (P/M) protocol including a ball-milling, molecular-level mixing, and friction stir processing, etc. [7–11]. Among the P/M process, the optimized properties of the Al/MWCNT composites fabricated by ball-milling process were as follows: elastic modulus of 75 GPa, yield strength of ∼450 MPa, and total elongation (TE) of 10% for the Al/3 vol% MWCNT composite [12–14]. Although the reports on the welding and joining of Al/MWCNT composites have facilitated their industrial application, their widespread applications were limited due to the poor weldability. The poor weldability was attributed to well-known welding defects (related to porosity, hot cracking, and high residual stress) along with clustering and/or decomposition of reinforcements during fusion welding [15–18], resulting in deterioration of mechanical properties. Unlike fusion welding, friction stir welding (FSW) leads to sound joints without significant degradation of mechanical properties, according to previous studies on $Al/Al_2O_3$ and Al/SiC composites [19]. These results can be attributed to the grain refinement due to dynamic recrystallization and homogeneous distribution of reinforcements by material mixing during FSW, which is a solid-state welding process involving both frictional heat and plastic deformation at relatively low temperatures [20].

The welding condition during FSW should be studied because the welding temperature can affect the reactivity between the reinforcement and the Al matrix [15,16,21,22]; unexpected reactions, such as the formation of aluminum carbide ($Al_4C_3$), can lead to the deterioration of mechanical properties depending on the characteristics of the $Al_4C_3$ [1,23]. When the formation of the $Al_4C_3$ is high temperature above the recrystallization temperature of the Al and a coarse $Al_4C_3$, there is a high possibility of causing repercussions in degraded mechanical properties. The welding and joining of the Al matrix composites is also difficult because of the low melting temperature and high thermal conductivity of Al matrix [24–27].

The research on the welding and joining of the Al-matrix composites is significantly important in both academic and industrial fields, however, it has not been reported yet. Therefore, in order to carry Al/MWCNT composites with outstanding mechanical properties to the level of industrial applicability, herein we report the application of FSW as an innovative solid-state process to produce sound joints with excellent balance between strength and ductility in Al/MWCNTs. In addition, this is the first time the relationship between the microstructure and mechanical properties of Al/MWCNTs after the FSW is elucidated by tuning both the plunging load and the amount of MWCNT.

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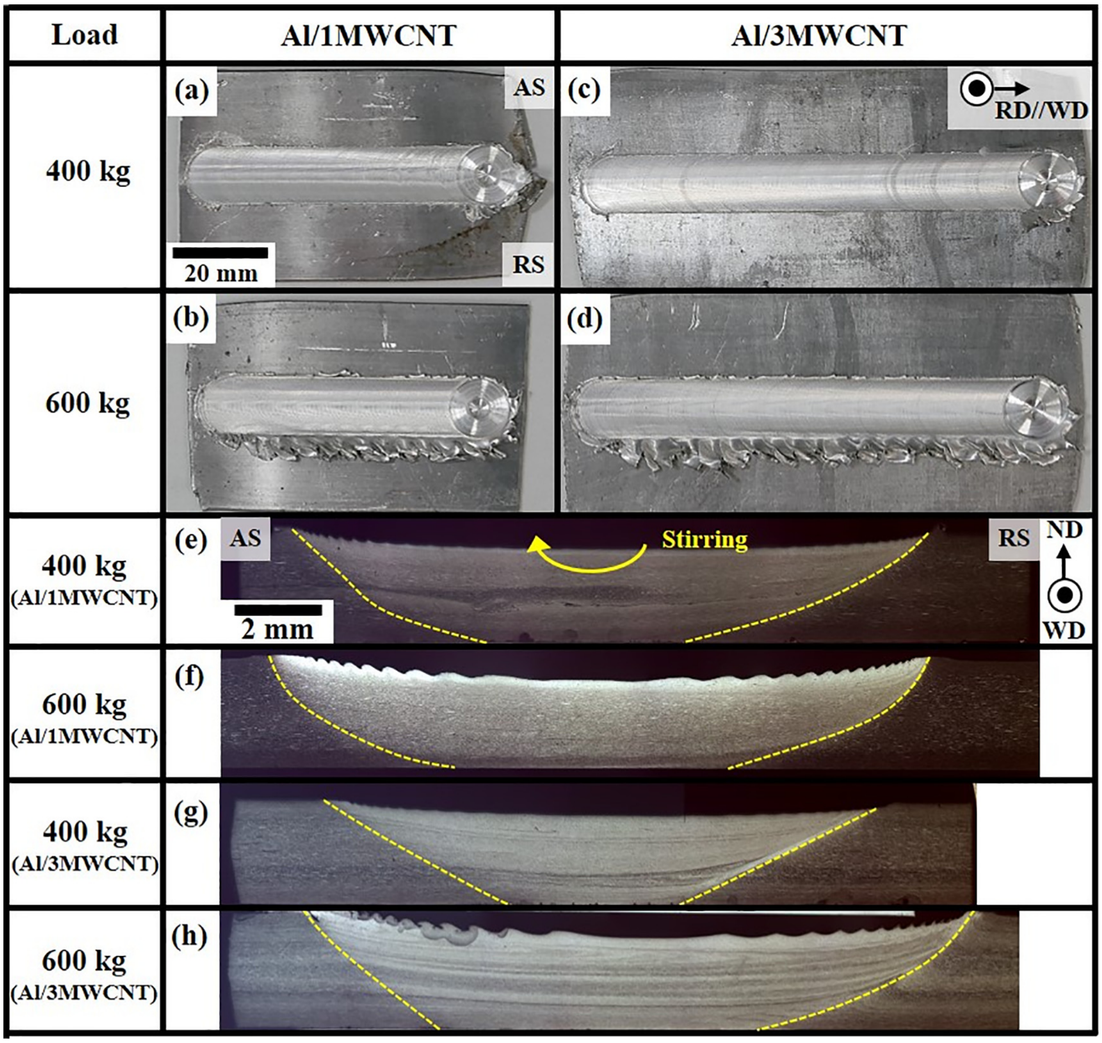


**Fig. 1.** FSWed Al/MWCNTs composite plates at the axial plunging loads of 400 and 600 kg under the constant welding speed of 200 mm min$^{-1}$ and rotation speed of 600 rpm; (a), (b) Al/1MWCNT and (c), (d) Al/3MWCNT. (e) OM images showing the cross-sectional macrostructures of the samples at the center and on the retreating and advancing sides (RS and AS, respectively). (RD: rolling direction, WD: welding direction, and ND: normal direction).

## 2. Experimental

### 2.1. Sample Preparation

The Al/MWCNT composite powders were fabricated by ball milling, using Al powder (mean diameter: 150 µm and purity: 99.5%) as the matrix, and 1 and 3 vol% MWCNTs (mean length: 5 µm, mean diameter: 10 nm, supplied from Applied Carbon Nano Co., Ltd.) as the reinforcement. The experimental details of the manufacturing process of the Al/MWCNT composite powder using ball-milling method in our pervious study [28]. The composite powders were hot-extruded to 50-mm-diameter rods at 400 °C with a 5:1 extrusion ratio, and subsequently warm-rolled at 350 °C with a thickness reduction of 10% per pass; the final thickness of the plate was ~2 mm. Since the MWCNTs were embedded inside the Al powder, they did not significantly interrupt the consolidation of Al powder during the hot-working. Hereafter, the Al/(1 and 3 vol%) MWCNTs are referred to as Al/1MWCNT and Al/3MWCNT, respectively.

### 2.2. Friction Stir Welding Condition

The warm-rolled plate, as the base metal (BM), was friction-stir welded (FSWed) in a bead-on-plate at plunging loads of 400 and 600 kg under the constant rotation speed of 600 rpm and traveling speed of 200 mm min$^{-1}$. A tungsten carbide rotating tool, with a 12.0-mm shoulder diameter, a 4.0-mm probe diameter, and a 1.8-mm probe length, was utilized with an advancing tilt angle of 3° from the normal direction. Argon was used as the shielding gas during the FSW, to protect the oxidation.

### 2.3. Measurement Method

The border between the welding zone and the BM of the FSWed Al/MWCNT was observed using an optical microscope (OM, Leica, ICC50, Germany) after etching by Keller's etchant (190 mL distilled water, 6 mL HCl, 5 mL $HNO_3$, and 2 mL HF) at room temperature for 90 s. The microstructures before and after the FSW were observed using a field-emission scanning electron microscope (FE-SEM; S-4300SE, Hitachi, Japan) equipped with an electron backscatter diffractometer (EBSD; EDAX-TSL, Hikari, Japan). The specimens for EBSD observation were sectioned, cold-mounted and ground using the abrasive papers up to 2000 grit, then mechanically-polished using the suspension including 0.04-µm colloidal silica for 30 min. The accelerating voltage, probe current, working distance, step size, and minimum confidence index for EBSD analysis were 20 kV, 12 nA, 15 mm, 0.06 µm, and 0.1, respectively. To measure the local misorientation, the Kernel average misorientation (KAM) value was calculated up to the 3rd neighboring shell with a maximum misorientation angle of 5° using the TSL-OIM