

# **Synthesis of MWCNT Reinforced Al-Si Nanocomposites by Microwave**

## **Sintering and Spark Plasma Sintering**

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### **Abstract**

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In the present study, first ever attempt has been made to develop physically functionalized multiwalled carbon nanotube (MWCNT) reinforced Al-11.5Si alloy nanocomposites synthesized via novel consolidation techniques, viz. microwave sintering (MWS) and spark plasma sintering (SPS). There is a recent trend in employing carbon nanotubes (CNTs), an allotrope of carbon, as reinforcement for high strength structural metallic composite materials, as these cylindrical nanofibers poses extremely unique mechanical properties such as very high elastic modulus (~ 300 GPa to 1.5 TPa) as well as tensile strength (~150 GPa). The mechanical strength of the CNT reinforced composite materials can further be improved by having nanometric grains in the matrix. However, it has remained as an ever-existing problem to achieve a porosity-free nanocrystalline matrix with homogenously dispersed CNTs, owing to the very high coagulation tendency of CNTs and non-availability of processing route to achieve or retain nanocrystallinity in the matrix.

Al-Si alloys are widely used in the aerospace and automobile application because of the high strength to weight-ratio, high specific stiffness and very good corrosion resistance of these alloys. The gas-atomized, spherical Al-11.5Si alloy powders (1-8  $\mu\text{m}$ ) were subjected to high energy ball milling for the purpose of achieving nanocrystallinity in the powders. The presence of nano-sized grains in the ball milled powder was confirmed from transmission electron

micrographs and X-ray diffraction study. The improvement in MWCNT dispersion was effected by treating the MWCNTs with a physical surfactant, sodium dodecyl sulfate (SDS). FEG-SEM micrographs of the mixture of SDS treated MWCNTs and ball milled Al-Si powders ensured homogenous dispersion of MWCNT. The nano-grained ball-milled Al-Si powders with varying MWCNT content (0.5 and 1 wt%) were consolidated via microwave sintering and spark plasma sintering in order to retain the nanosized grains in the Al-Si matrix, attributed to the faster and highly effective sintering kinetics of these two sintering techniques. Consolidation of the mixture of ball-milled Al-Si powder and MWCNTs was also carried out by conventional hydrogen furnace sintering for comparison purpose.

The morphology and densification of the various nanocomposites were studied by optical and scanning electron microscopy, and quantitative image analysis. The best densification (97%) was achieved for 0.5 wt% SDS treated MWCNT reinforced SPS nanocomposite, owing to the better dispersion of lower weight percent physically functionalized MWCNTs and better sintering kinetics of SPS. Inter-particle boundaries were observed in MWS nanocomposites with non-SDS treated 0.5 and 1 wt% MWCNTs and in less occurrence in SPS nanocomposite with non-SDS treated 1 wt% MWCNTs. However, in case of using SDS treated MWCNTs as reinforcement, the inter-particle boundaries were only visible for MWS nanocomposite with 1 wt% MWCNTs.

The TEM and XRD study confirmed the presence of nano-sized grains in the MWS and SPS nanocomposites. Detailed TEM study of the 0.5wt% MWCNT reinforced SPS nanocomposite revealed that the movement and arrangement of dislocations in order to cause grain refinement, which happened during high energy ball milling was retained in the Al-Si matrix of the nanocomposite. Moreover, presence of low angle grain boundaries was also observed in this nanocomposite indicating this phenomenon to occur during ball milling. Presence of primary Si

particles were observed at the grain boundaries in the matrix of this nanocomposite, providing resistance to grain coarsening. Besides, excellent adherence between the MWCNT and Al-Si matrix was also envisioned from the HRTEM micrographs.

Hardness and elastic modulus measured by nanoindentation technique, were observed to be improved with using lower content (0.5 wt%) of surfactant treated MWCNT reinforcement in the nanocomposite synthesized by SPS. Nanoscratch was carried out for evaluating the tribological property, which revealed that the volume of material loss is least in the SPS nanocomposites, compared to that in the nanocomposites sintered by the two other methods. Variation of Coefficient of friction (COF) values with the scratch distance which was attributed to the microstructural irregularities was observed to be least in case of SDS treated 0.5 wt% MWCNT reinforced SPS nanocomposite.

**Keywords:** Nanocomposite, Al-Si alloy, MWCNT, ball milling, physical functionalization, microwave sintering (MWS), spark plasma sintering (SPS)