



# Nanoscratch behavior of carbon nanotube reinforced aluminum coatings

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## ABSTRACT

Nanoscratch experiments have been carried out on plasma sprayed aluminum alloy coatings reinforced with 0, 5 and 10 wt.% carbon nanotubes (CNTs). Scratches have been performed at loads of 1000, 2000 and 3000  $\mu\text{N}$  load using Berkovich indenter. The contact and true wear volumes of the scratches have been calculated. The nano-scale wear resistance is shown to increase by 4 times by addition of 10 wt.% CNTs. Improvement in the wear resistance is discussed with respect to strengthening effect and increased elastic recovery by addition of CNTs. Direct evidence of increased recovery and small decrease in the coefficient of friction with CNT content is provided using the true and instantaneous depth plots and the corresponding scanning probe microscope and scanning electron microscope images of the scratches. Friction coefficient was found to be load independent and was found to vary slightly with the CNT content.

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## 1. Introduction

Nanoscratch testing using a nanoindenter is a unique method for characterizing the wear properties of a surface at the nano-scale. This kind of testing is significant for predicting the wear behavior in case of micro- and nano-electro mechanical devices [1] employing moving parts like gears and applications involving thin coatings like magnetic storage devices [2]. Also it helps in understanding processes in which nano-scale patterning is carried out by removal of material. Nanoscratch experiments have been carried out to study the wear behavior of different kinds of materials like polymer/polymer composites [3–6], hard ceramics [7–10] and diamond-like carbon coatings [11–13], self lubricating transition metal dichalcogenides [14], metals [15–17] and human hair [18]. The focus of these studies has been to understand the near-surface wear mechanisms, deformation and damage in the wake of the indenter, critical load for delamination and adhesion strength of thin films, effect of reinforcement on the wear resistance or understanding the friction behavior of the coatings. Dasari et al. [4] have used nanoscratch studies on nylon 66-SEBS-organoclay nanocomposites to understand the effect of dispersion of exfoliated clay between the hard and soft regions on the nanoscratch properties. Yuan et al. [5] have observed the formation of ripple type tracks during nanoscratching of polypropylene-clay composites which were analogous to periodic ripples formed during micro-scratching at larger loads. This suggested that the mechanism of microscale deformation could be applied at the nano-

scale. An in-depth stress analysis around the indenter was presented by Ghosh et al. [10] to show that a maximum shear stress occurred in front of the indenter and maximum principal tensile stress in the wake of the indenter. These stress distributions were responsible for the observed slip bands and microcracks on the wear track. Lafaye and Troyon [16] have analyzed the variation of friction force with the applied normal force and provided a model based on the elastic recovery. Wei and Bhushan have used the nanoscratch technique to study the properties of human hair treated with different conditioners and shampoos [18]. It was mentioned that these studies may help in developing or testing hair care products. Thus, nanoscratch technique is very important technique and the interest and understanding of this technique is rapidly growing.

Carbon nanotubes (CNTs) have been proposed as reinforcement for polymer, ceramic and metallic matrices due to their excellent mechanical properties [19–23]. Balani et al. have shown tremendous improvement of wear resistance of alumina coatings reinforced with CNTs both at nano (18 times) and macroscale (49 times) [8]. Excellent improvement in the mechanical properties has been reported for Al [23–29], Cu [30,31] and Ni [32,33] matrix composites. Zhou et al. [29] have shown a 30% decrease in the wear rate and friction coefficient of Al-CNT alloys by addition of 20 vol.% of CNTs. More than 200% increase in the yield strength has been observed in 10 vol.% CNT reinforced Cu composites prepared by a molecular level mixing method which shows the significance of dispersion in effective strengthening [31]. A decrease in the macroscale wear loss by 80% and coefficient of friction by 40% by addition of CNTs was observed in Ni-P-CNT coatings prepared by electroless deposition technique [33]. These results indicate that CNT addition could result in improvement in mechanical and wear properties.

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The goal of this paper is to study the effect of CNT content on nano-scale wear resistance of plasma sprayed aluminum–CNT composite coatings. Nanoscratch testing leads to study of the reinforcement phenomena by CNTs at the nano-scale and the effect on the friction. The study of individual scratches provides vital information regarding the mechanisms of enhancement of wear resistance by CNT addition. Also the mechanism of scratch damage is of interest in fundamental understanding of macro wear phenomena. There are no studies on nano-scale wear behavior of CNT reinforced metal matrix composites.

## 2. Experimental techniques

### 2.1. Coating fabrication

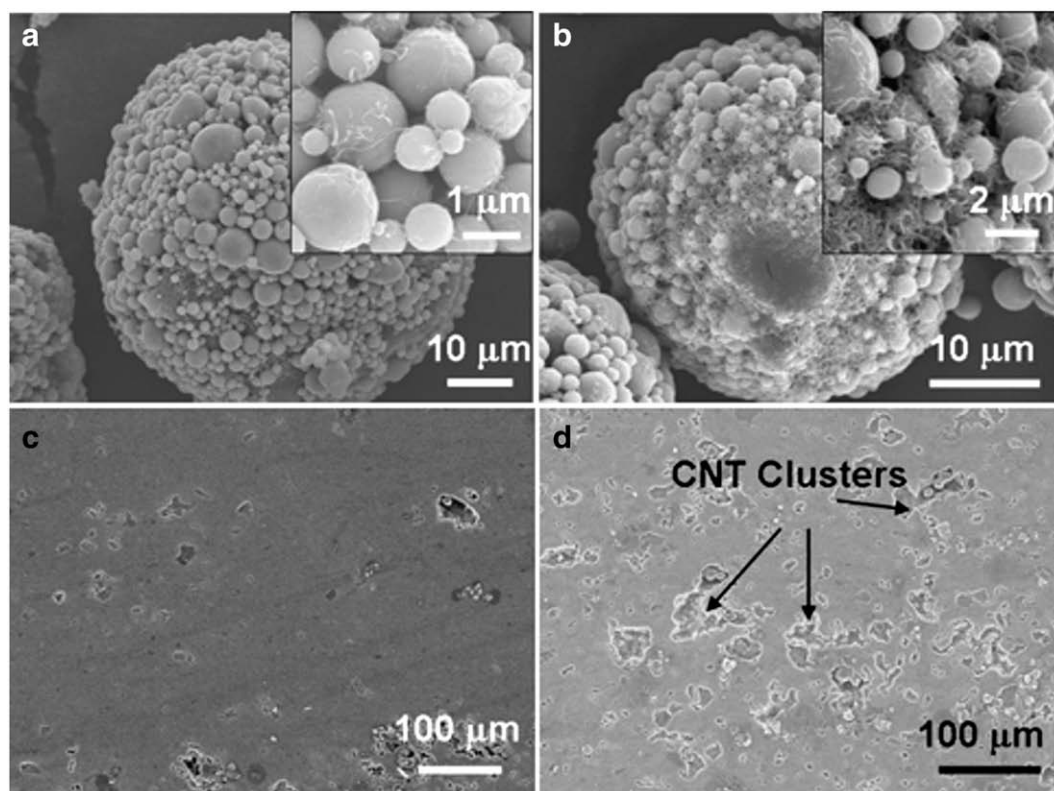
The feedstock powder used in this study was prepared by subjecting a slurry of 1–3  $\mu\text{m}$  size inert gas atomized Al–11.6 wt% Si alloy (referred as Al–Si hereafter) powders containing carbon nanotubes (CNTs) to spray drying. The carbon nanotubes used were more than 95% pure having a diameter of 40–70 nm and length of 1–3  $\mu\text{m}$ . Spray drying resulted in spherical agglomerates of Al–Si particles with CNTs uniformly distributed on the surface of the particles. Powders of two compositions containing 5 wt.% and 10 wt.% CNT were made. Aluminum–silicon coatings reinforced with CNTs were prepared by plasma spraying of the spray dried powders onto a mild steel substrate. During plasma spraying, the powders are injected into an inert gas plasma created between an arc between a tungsten cathode and a concentric copper anode, where they get molten/superheated and are accelerated towards the substrate to impact and deposit as splats. Coating results from a layer by layer deposition of such splats. The baseline coating of Al–11.6 wt.% Si alloy was made using gas atomized powders of size  $14 \pm 9 \mu\text{m}$  for the purpose of comparison. The coatings with 0, 5 and 10 wt.% CNT will be referred to as Al–Si, Al–5CNT and Al–10CNT hereafter. Coatings of up

to 1500  $\mu\text{m}$  were made details of which have been reported elsewhere [28].

### 2.2. Nanoscratch testing and imaging

The coating cross section was mounted using epoxy and ground up to 600 grit size emery paper followed by metallographic polishing with diamond suspension up to 0.1  $\mu\text{m}$  size. Nanoscratch testing was carried out on the polished cross sections using a Triboindenter (Hysitron Inc., Minneapolis, USA). It has a horizontal capacitive transducer for applying normal load and two vertical capacitive transducers for measuring the lateral force experienced by the indenter during scratching. The transducers measuring the lateral force have a load resolution of 3  $\mu\text{N}$  and a normal displacement resolution of 0.04 nm. Scratches of length 20  $\mu\text{m}$  were made at loads of 1000, 2000 and 3000  $\mu\text{N}$  using a Berkovich tip. During the loading cycle, the indenter moves 10  $\mu\text{m}$  to one side of the mean position after which the load is applied. During this movement the indenter records the surface profile from which the tilt of the sample is measured. The correct instantaneous depth is obtained by subtracting the tilt from the measured values. After the load has reached the set value, the indenter starts scratching at a speed of  $0.67 \mu\text{m s}^{-1}$ . When the scratch length has reached 20  $\mu\text{m}$ , the load is released. The Berkovich tip is in the form of a triangular pyramid with total included angle of  $142.3^\circ$  and has a tip of radius of curvature equal to 100 nm. After the scratching has been performed, the same tip was used to image the surface by applying a contact load of 2  $\mu\text{N}$ . The resultant scanning probe microscopy (SPM) images were analyzed using the SPM image processing software SPIP<sup>TM</sup> (Image Metrology A/S, Horsholm, Denmark). Depth profiles were taken along lines parallel and perpendicular to the scratch using SPIP<sup>TM</sup>.

In order to examine the wear scratch and correlate the wear properties to the local microstructure, SEM was carried out on the scratched coatings. The scratches were also examined using a JEOL



**Fig. 1.** SEM micrographs of a) and b) spray dried Al–5CNT and Al–10CNT powder respectively powder with inset showing a high magnification picture of the powder, and c) and d) plasma sprayed Al–5CNT and Al–10CNT coating respectively.

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