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## Feature article

## Plasma sprayed carbon nanotube reinforced splats and coatings



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#### ABSTRACT

Plasma sprayed composite coatings prepared from powders doped with 1 wt% CNT were characterized by their scratch resistance. Additionally, the scratch resistance of the splats obtained from one powder, namely, crushed alumina, was also investigated. The average splat diameter of the splat containing CNT was larger. CNT reinforced splats were more damage resistant as compared to those produced from the pristine powder. The estimated increase in the work of adhesion, while scratching the CNT reinforced coatings was 59–176% higher than their counterpart without CNT. This is attributed to CNT bridging across splats resulting in an improvement in coating properties. Other factors promoting coating adhesion were CNT anchorage in the asperities of the bond coat and enhancement of coating stiffness in the presence of CNT. In general, coating failure during scratching occurred owing to the formation of tensile crack and large area spallation ahead of the indenter.

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#### 1. Introduction

Plasma sprayed coatings are constituted by deformed frozen particles known as splats [1,2]. Alumina coatings deposited by plasma spraying are known for their high hardness and wear resistance [1,3]. Sabiruddin et al. [4] reported a hardness of around 1200  $HV_{0.3}$  for such coatings. Plasma sprayed titania coating is another example of wear resistant coatings.

The scratch test is a well-known method for estimating the adhesive strength of thin coatings [5]. Very few reports are available on the scratch resistance of thermally sprayed coatings. During scratch test, a thermally sprayed coating may fail in cohesion, adhesion or both. One common failure mode of such coating is by formation of tensile cracks normal to the scratching direction [6].

Carbon nanotube (CNT) possess high tensile strength and elastic modulus and thus, are well suited as reinforcing element in a composite [7,8]. A thorough investigation on plasma sprayed CNT reinforced alumina composite coatings has been undertaken by Agarwal and his team [9,10]. They made the following important observations. (1) Alumina melts in-flight and creates a protective sheathing on the CNT elements. Thus, a fraction of the CNT is protected from the plasma environment during deposition [11].(2) The hardness of a 1.5 wt% CNT reinforced coating is around 15% higher than its conventional counterpart [12]. (3) Addition of 8 wt% CNT

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http://dx.doi.org/10.1016/j.jeurceramsoc.2017.01.028 0955-2219/© 2017 Elsevier Ltd. All rights reserved. reinforcement produced an increase in indentation fracture toughness from  $3.22 \pm 0.22$  to  $5.04 \pm 0.58$  MPam<sup>0.5</sup> in a plasma sprayed alumina coating [10]. In all these investigations a He – 33 Vol% Ar gas mixture was used [13,14]. Also, for most of these experiments the powders were prepared by mixing 1.5–8 wt% CNT in alumina either by mechanical mixing or spray drying [9,15].

Hazra and Bandyopadhyay [16] investigated the scratch resistance of conventional and nano-structured alumina coatings using a Rockwell C type indenter. Tensile cracks appeared in the coating during scratch test. In most of the cases, coating failure was attributed to large area spallation.

As stated, CNT tend to form agglomerates, and a powder containing such agglomerates is unsuitable for spraying. Agarwal and his group used the spray drying technique to disperse CNT in alumina [9]. Heterocoagulation, a colloidal processing technique, can serve as an alternative means to ensure a homogenous distribution of CNT in a powder [17]. Preparation of micro-sized, CNT reinforced, freely flowing plasma spray ceramic feedstock using heterocoagulation was introduced by Jambagi et al. [18].

This document is one of the first reports on the composite coatings produced from the above powders. This work differs significantly from those of the predecessors on two counts. First, the composite coatings were deposited using low cost nitrogen-10 vol% hydrogen mixture. This gas is used widely in the industry owing to its lower cost and easier availability as compared to helium or even argon. Second, the CNT concentration in this investigation was limited to 2 wt%.

The objective of this work is to investigate a few plasma sprayed ceramic composite coatings prepared from ceramic powders doped with CNT using heterocoagulation technique and characterize such coatings and splats produced from one powder in terms of their scratch resistance. The performance of these coatings has been compared with the same material not containing CNT. To the knowledge of the authors, it is the first attempt to characterize a CNT reinforced coating by measuring their scratch adhesion strength.

#### 2. Experimental procedure

# 2.1. Processing of powders, splats, coatings and the primary coating characterization

Three types of powders were used in this investigation, namely, agglomerated alumina, crushed alumina and crushed titania. The powders were deposited either in the as-received state or after doping with 1-2 wt% CNT. CNT doping was undertaken using heterocoagulation, a colloidal processing technique. In this process the two components of the mixture, namely, ceramic and CNT were taken in water in two separate containers and endowed with two opposite surface charges by adding appropriate surfactants. For the ceramic powders and CNT, anionic Sodium dodecyl sulfate (SDS) and cationic cetyl trimethyl ammonium bromide (CTAB) surfactants were used, respectively. In the presence of the surfactants the solids were suspended in water. These two suspensions were slowly mixed. During mixing, the solids with opposite surface charges contacted each other, lost their surface charges and settled to the bottom of the container. This mixture was filtered and dried to form a plasma spray feedstock. In a heterocoagulated powder CNT was found to be uniformly distributed in the ceramic. The powder preparation procedure has been discussed in details

Table 1

Powder and	coating	nomenclature.
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Coatings	Description
Coatings without CNT HA0 HC0 HT0	Agglomerated alumina without CNT Crushed alumina without CNT Crushed titania without CNT
Heterocoagulated HA1,HA2 HC1,HC2 HT1, HT2	Agglomerated alumina with 1 or 2 wt% CNT Crushed alumina with 1 or 2 wt% CNT Crushed titania with 1 or 2 wt% CNT

in a previous publication [18]. The coatings were deposited on C20 steel substrates using a 40 kW plasmatron (Metco 3MB, Westbury, NY, USA) mounted on a CNC XY table. The main parameters used for coating deposition using both types of alumina (Table 1) and titania powders are as follows: arc current 400 A, voltage 60-64 V, primary gas (nitrogen) flow rate 50 slpm, secondary gas (hydrogen) flow rate 5 slpm, powder federate 1.5 kg/h, standoff distance 125 mm. The coating deposition procedure has been discussed in details in a previous publication [19]. Powders and coatings used in this investigation and their nomenclature are listed in Table 1. Splat deposition was undertaken using crushed alumina powder doped with 2 wt% CNT and these splats are designated as HC2. The procedure of splat collection is as follows. A zirconia coated steel curtain with a hole of size 6 mm was held at a distance of 75 mm from the front end of the gun exit and the hole was at an offset of 5 mm vertically downward from the plasma jet axis. This perforation in the curtain was aligned with central line of the stream of molten particles coming out from nozzle, taking gravity into account. The round substrate was held in tong and preheated



Fig. 1. Optical micrograph of crushed alumina splats on polished bond coat, (b) magnified image of an isolated splat enclosed by the rectangle in (a), and (c) the histogram showing splat size analysis.

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