Result of laser ablation and flame test on SiC, Graphene, CNT coated AISI 321

J. Daniel Glad Stephen*, G.M. Brahmanandhan, Aranav Das, M. Syed Zameeruddin
Department of Mechanical Engineering, SRM University, Kattankulathur – 603 203
***Corresponding author: E-Mail: jdgstephen@gmail.com**

ABSTRACT

With the increasing requirement for air transport, there is a demand for more efficient, safe aircraft. This project concentrates upon improving the thermal stability of ceramic coatings which is one of the most common coating material used in the aircraft nozzle. Since ceramics have low toughness at high temperature, other materials are added to the ceramic to enhance its toughness. One of the best materials is SiC, Graphene, and CNT because of their high temperature stability and high melting point. A mixture of SiC powder with commercially available single walled CNT is synthesized. A coating of reduced Graphene oxide is made in AISI 321, on which the mixture of silicon carbide and SWCNT is coated. Then the coated samples have been subjected to Laser ablation test and flame test. The laser ablated, and flame tested samples have been subject to SEM, EDX analysis for finding out the crack development. From the SEM images, it is evident that the coating formed has a matrix of Graphene and Silicon Carbide and F-SWCNT sandwiched together layer by layer and there were no significant cracks have been developed.

KEY WORDS: SiC, Graphene, CNT, SEM, EDX, Laser ablation.

1. INTRODUCTION

The coating is a material processing technique to cover a substrate to enhance the performance of materials like AISI 321. There are a lot of coating methods are available. In all the methods the preliminary objective is to coat the material on some substrate (Yunfeng, 1997).

Atmospheric plasma spraying (APS), which is a high-energy coating process with plasma temperatures in the region of 20,000 K and powder particle velocities of up to 450m/s. The powder which is suspended in a carrier gas was injected into the plasma. After melting and being accelerated, the powder particles impacted the substrate and built up the coating (Fauchais, 2004). Pre-ceramic precursor derived ceramic technology is used to fabricate ceramic coatings at low temperatures. The precursor could be cured to form a polymer coating at room temperature by UV light or at temperatures below 373 K with thermal initiator (Xiang Yang, 2014; Nangrejo, 2000). The slurry method is one technique, which could be used to fabricate coatings on complex shaped components at low cost. This technology required very high heat-treatment temperature to obtain dense coatings. The high temperature could damage the composites to some extent (Xiang Yang, 2014; Wang, 2006). Reactive preparation is a technique, which could be used to fabricate coatings by a reaction. For examples, ZrB_2 coatings upon C/SiC composites were prepared by pasting the slurry of zirconium powder, boron powder, and phenolic resin, followed by high temperature sintering (Nath, 1980). Sol–gel method, reported in recent literature (Kiyoharu Tadanaga, 2000), was an easy way to produce oxidation-resistant coatings at low processing temperatures. ZrO_2-SiO_2 gradient multilayer oxidation protective coatings were prepared by a sol–gel technique using Tetra Ethoxysilane (TEOS) and Zirconyl Chloride as source materials. Chemical vapour deposition is one of the most popular solutions for coating technology. CVD was a synthesis process: the chemical constituents react in the vapour phase near or on a heated substrate to form a solid deposit. This technology is used in engineering disciplines including thermodynamics, plasma physics, kinetics, fluid dynamics, and of course chemistry (Gary, 1988). Microwave sintering, as a novel and effective sintering method for ceramics, was adopted to prepare the coatings for C/SiC composites. Molten salt reaction method is a technique, which could be used to fabricate coatings on complex shaped components at low cost and low temperature. Whatever may be the coating method, the objective of the coating is to enhance the surface morphology, mechanical, chemical and anti-corrosion property (Anklekar, 2001).

AISI 321 is a aircraft parts manufacturing material usually used in manufacturing the nose component and turbine blades, has to withstand lot of thermal stress and strain. To increase its mechanical properties especially thermal properties, ceramics such SiC or CNT usually coated (Zhidong Han, 2011). Because of the coasting, the AISI 321 can withstand lot of thermal stress and strain. So, in this project, the AISI 321 has been selected as substrate and SiC + Graphene + CNT has been chosen as the coating materials and the results are discussed.

2. EXPERIMENTAL METHODOLOGY

Even though, a lot of coating methods are available, for the coating of Graphene spin coating has been used. Spin coating is a technique where the coating has been done very effectively and in a very easy manner. The Graphene chosen for this work is having a Purity of 99%, 5-10nm size. SiC is also 99% pure having a particle size of 25-50nm and the final coating material CNT is a Single Walled Carbon Nano Tube having a particle size of <10nm is used.

Base Metal Preparation: The base metal used in this work is AISI 321. This alloy is selected because it is used to produce certain high temperature components of the turbine engine. The substrate was grinded to 5mm thickness and then buffed with large grain sandpaper to attain a roughness of about 50µm. The substrates were then etched in dilute HCl solution for about an hour.

Ultra sonication Process:

CNT: In a typical procedure, 0.5 g of pure SWCNTs were treated with a 3:1 mixture of concentrated $H_2SO_4$and HNO_3, and then the mixture was sonicated at 40^0C for 3 h in an ultrasonic bath. After cooling to 27^0C, the mixture was diluted with 500 mL of de-ionized water and then vacuum-filtered through a filter paper of 3µm porosity. The resultant solid filtrate (f-SWCNTs) was repeatedly washed with deionized water until the pH became neutral and then dried in vacuum at 60^0C for 10 h.

Graphene: The same Ultra sonication process was used for r-Go mixture but at low frequency. Initially 0.05gm of reduced Graphene oxide is dispersed in 20ml of DI water and sonicated for 2h. Then 2gm of PVP is added to the sonicated mixture and sonicated at 80^0C for 5 h with constant stirring. The system is immersed in an oil bath to maintain a constant temperature.

Spin Coating: The procedure followed was very simple. A sample substrate of size 1*1 cm is placed inside the ceramic chamber of the spin coater. An earlier prepared colloidal solution of Graphene is used for the coating purpose. Using a 750 µL micro syringe, the solution was drop casted on the substrate. Then the machine was allowed to run for 60 seconds at 2000rpm at room temperature. Due to the centrifugal force, the solution was evenly spread upon the substrate resulting in an even coating (Vincent, 2009). This coating was then dried in a hot air oven at 80^0C for 20 minutes and then the coated materials very kept inside a muffle furnace. The objective to use the muffle furnace was to heat the coated samples at elevated temperatures for adherence of the particles of SiC with CNT. Finally SiC + Graphene + CNT coated AISI 321 is prepared for coating.

Laser Ablation and Flame Test: The tests were carried out to determine the strength and the durability of the coating at high temperatures (Ryan, 2003). TBC-deposited specimens were laser-treated by a continuous CO_2 laser (max 3.5 kW, PLR Laser). The diameter of the laser spot was 1.2 mm. By defocusing the spot, the effective laser area was widened to 200 mm^2. The power of the laser was maintained 500 W for about 10 seconds for each sample. After the laser ablation test, the flame test was carried was carried out on the ablated samples. For the flame test, the samples were subjected to a gas flame which instantaneously increased the temperature for about 1minute per sample. After that treated and untreated SiC + Graphene + CNT coated AISI 321 has been characterized by SEM and EDX spectroscopy.

3. RESULTS AND DISCUSSIONS

This laser ablated and flame tested samples is subjected to FESEM analysis for the surface morphological measurement. The FESEM images of laser ablated with or without flame tested samples have been given in the figure 1. The Figure 1a is the FESEM of laser ablated SiC + Graphene + CNT coated AISI 321, shows little cracks in the nano meter range. But in figure 1b, the flame tested samples of same laser ablated coated materials are not having any cracks, since the flame tested material's grain sizes increases due to that the cracks are not developed.

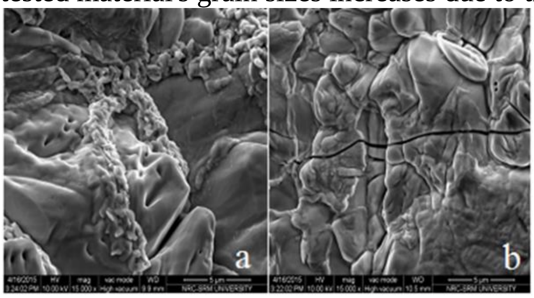

Figure.1. FESEM of a) laser ablated and flame tested and b) laser ablated SiC + Graphene + CNT coated AISI 321

The EDX images of the laser ablated and flame tested sample of SiC + Graphene + CNT coated AISI 321 is given in figure.2. From the EDX images for laser ablated and flame tested SiC + Graphene + CNT coated AISI 321, the presence of oxygen, iron and silicon have been identified as (51, 41, 7)% and there were no observable impurities (Jacek Ryl, 2014). The tables.1 and 2 shows EDX measured composition of laser ablated and flame tested SiC + Graphene + CNT coated AISI 321 material.

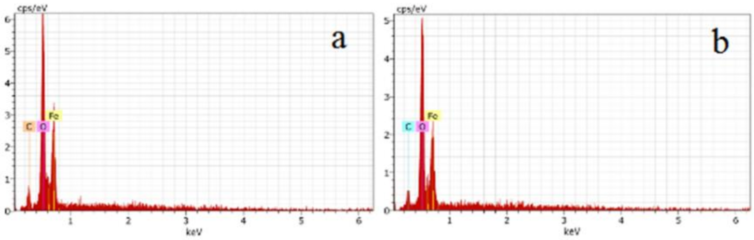

Figure.2. EDX image of a) laser ablated and flame tested and b) laser ablated SiC + Graphene + CNT coated AISI 321

Table.1. Edx data of laser ablated and flame tested SiC + Graphene + CNT coated AISI 321 material

El	AN	Series	unn. C (wt. %)	norm. C (wt. %)	Atom. C (wt. %)	Error (1 sigma) (wt. %)
O	8	K - series	43.49	51.11	70.10	7.62
Fe	26	K - series	35.25	41.43	16.28	3.32
C	6	K - series	6.35	7.46	13.63	2.31
Total			85.08	100	100	-

Table.2. Edx data of laser ablated Sic + Graphene + CNT coated AISI 321 material

El	AN	Series	unn. C (wt. %)	norm. C (wt. %)	Atom. C (wt. %)	Error (1 sigma) (wt. %)
Fe	26	K - series	50.64	51.81	22.80	3.91
O	8	K - series	41.06	42.01	64.55	6.94
C	6	K - series	6.04	6.18	12.65	2.10
Total			97.74	100	100	-

From the FESEM images and EDX measurement, it is evident that the absence of cracks and absence of impurities the mechanical properties especially thermal properties of the AISI 321 is enhanced.

4. CONCLUSIONS

Based upon the samples analysed, the following conclusions have been drawn-

- Samples subjected to both laser ablation and flame test showed resistance to cracks and specifically samples with CNT combination based on the SEM analysis.
- Sample when subjected to laser ablation showed microcracks. But upon flame test the samples showed a very minimal cracks.
- EDX analysis confirms that specific carbon content is present for each combination of coating as predicted earlier. Hence the AISI 321 with coating of SiC + Grpahene + CNT showed better resistance to crack formation.

REFERENCES

Anklekar R.M, Agrawal D.K & Roy R, Microwave sintering and mechanical properties of PM copper steel, Powder Metallurgy, 44 (4), 2001, 355-362.

Fauchais P, Understanding plasma spraying, Journal of Physics D, Applied Physics, 37, 2004, 9.

Gary W, Rice, Richard L, Woodin, Zirconium Borohydride as a Zirconium Boride Precursor, Journal of the American ceramic Society, 71 (4), 1988, 181-183.

Jacek Ryl, Anna Arutunow, Mateusz T, Tobiszewski, Joanna Wysocka, Aspects of inter granular corrosion of AISI 321 stainless steel in high-carbon-containing environments, Anti-Corrosion Methods and Materials, 61 (5), 2014, 328–333.

Kiyoharu Tadanaga, Junichi Morinaga, Atsunori Matsuda, Tsutomu Minami, Super hydrophobic−Super hydrophilic Micropatterning on Flowerlike Alumina Coating Film by the Sol−Gel Method, Chem. Mater, 12 (3), 2000, 590–592.

Mikhutkin A.A, Vasiliev A.L, Three-dimensional reconstruction of a surface based on scanning electron microscopy images, Crystallography Reports, 59 (6), 2014, 908-915.

Nangrejo M.R, Xujin Bao, Mohan J, Edirisinghe, Preparation of silicon carbide–silicon nitride composite foams from pre-ceramic polymers, Journal of the European Ceramic Society, 20 (11), 2000, 1777–1785.

Nath P, Bunshah R.F, Preparation of In_2O_3 and tin-doped In_2O_3 films by a novel activated reactive evaporation technique, Thin Solid Films, 69 (1), 1980, 63-68.

Ryan T, Kelly, Adam T, Woolley, Thermal Bonding of Polymeric Capillary Electrophoresis Microdevices in Water, Anal. Chem, 75 (8), 2003, 1941–1945.

Vincent C, Tung, Matthew J, Allen, Yang Yang, Richard B. Kaner, High-throughput solution processing of large scale grapheme, Nature Nanotechnology, 4, 2009, 25 - 29.

Wang X, Lan W.H, Xiao P, Fabrication of yttria stabilized zirconia coatings by a novel slurry method, Thin Solid Films, 494 (1–2), 2006, 263–267.

Xiang Yang, Chen Zhao-hui, Cao Feng, High-temperature protective coatings for C/SiC composites, Journal of Asian Ceramic Societies, 2 (4), 2014, 305–309.

Yunfeng Lu, Rahul Ganguli, Celeste A. Drewien, Mark T, Anderson C, Jeffrey Brinker, Weilang Gong, Yongxing Guo, Hermes Soyez, Bruce Dunn, Michael H. Huang & Jeffrey I. Zink, Continuous formation of supported cubic and hexagonal mesoporous films by sol–gel dip-coating, Nature, 389, 1997, 364-368.

Zhidong Han, Alberto Fina, Thermal conductivity of carbon nanotubes and their polymer nanocomposites: A review, Progress in Polymer Science, 36 (7), 2011, 914–944.