Effect on Surface Roughness of Laser Ablated SS 321 Material Coated with Graphene and SiC

J. Daniel Glad Stephen* and G. M. Brahmanandhan
Department of Mechanical Engineering, SRM University, Kattankulathur - 603203, Tamil Nadu, India; jdgstephen@gmail.com, g.m.brahma@gmail.com

Abstract

Objectives: SS 321 is an austenitic stainless steel material which finds application in many fields. The coating on SS 321 by Graphene and SiC are used to improve the thermal resistance and strength of SS 321. Methods/Statistical Analysis: Coating of Graphene and SiC on SS 321 was done by spin coating and drop casting. The coated samples were subject to laser ablation followed by flame treatment. Then treated and untreated samples were characterized by SEM, EDX and Surface roughness measurement. Findings: From the SEM images the coating was found to be uniform and from the EDX measurement, the composition of the samples has been calculated and no observable impurities found. From the surface roughness measurement, it is evident that the roughness parameter changes from untreated to treated samples but there were no sign of cracks, which proves that the coating by Graphene and SiC on SS 321 is viable and increases the strength of the material. Application/Improvement: By doing Atomic Force Microscopy and thermal resistance and strength measurements on Graphene SiC coated SS 321 can give further authentic result or by choosing CNT like materials for coating, may improve the performance of SS 321.

Keywords: Graphene, SS 321, SiC, Spin Coating, Surface Roughness, SEM

1. Introduction

SS 321 is an austenitic stainless steel, which has chromium-nickel alloy to which titanium has been added, so it is favourable candidates for use at elevated temperatures, and it has high strength, excellent toughness, good corrosion resistance, weldability, etc. Since this material can be used at elevated temperature, the useful environments for this material exceeds 773K or reaches up to 1173K1. Molybdenum has also added to improve the corrosion resistances. The composition of SS 321 is given in Table 12. As it is an AISI material, mostly it can found in the nose of the airplane where it is expected to have a high thermal environment. Also in manufacturing the turbine blades, SS 321 finds a place.

Table 1. The composition of SS 321

<table>
<thead>
<tr>
<th>COMPOSITION</th>
<th>(wt %) max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.08</td>
</tr>
<tr>
<td>Manganese</td>
<td>2.00</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.045</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.030</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.75</td>
</tr>
<tr>
<td>Chromium</td>
<td>17.00 – 19.00</td>
</tr>
<tr>
<td>Nickel</td>
<td>9.00 – 12.00</td>
</tr>
<tr>
<td>Titanium</td>
<td>5 x (C + N) min., 0.70 max</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>0.10</td>
</tr>
<tr>
<td>Iron</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The coating on a material such as SS 321, usually used to improve the surface properties, weldability,
corrosion resistance and adhesion. A lot of coating methods such as Atmospheric plasma spraying, Chemical vapour deposition, thermal evaporation, spin coating and dip coating, etc are done on materials. Graphene is a new generation material, an allotrope of carbon element. Due to the unique structure, it has high electronmobility at room temperature. It has excellent mechanical property having a young’s modulus of 1TPa and tensile strength of 130GPa. In terms of thermal properties, the highest thermal conductivity at room temperature was reported as 5000 Wm−1K−1. Because of these properties, it finds application in Gas detection, transistors, nano-composites, energy storage devices, barrier applications and so on. Because of its physical, chemical and mechanical properties Graphene, is one of the material which can enhance the performance of substrate material such as SS upon coating. Graphene was also proved to be an effective corrosion barrier material because it was considered inert under the conditions where chemical reactions of other substrates will take place.

The Thermal Barrier Coating (TBC) using materials such as aC or SiC composites increases the working temperature of the SS 321. By coating the C/SiC on SS 321 is expected to increase the thermal stability of the stainless steel.

Corrosion is a process that is related to both environmental factors and the conditions of the metal surface. Although stainless steel is more resistant to corrosion than other metals, in acidic, aqueous environments, it can be susceptible to localized forms of attack that can result in cracking. By coating Graphene on SS 321 usually improves the corrosion resistance, without compromising the properties or structure of the native stainless steel material.

In this paper, the result of Graphene and SiC coating on SS 321 has been presented. The Graphene and SiC coating are chosen as the coating material for increasing the thermal stability and corrosion resistance of the SS 321.

2. Materials and Methods

99% purity Graphene with a particle size of 5-10 mm has been chosen for the coating materials. While the substrate is SS 321. The SS 321 is single walled having a particle size of 25-50 µm has been chosen for this study. Ultrasonicator machine is used for creating chemical solutions of Graphene, and SiC, and then Spin Coater is used for coating Graphene on SS 321 substrate. Then the coated samples were kept in Muffle Furnace for coating SiC after that laser ablation and flame treatment has been performed. Surface Roughness profile measurement has been done on the coated samples.

2.1 Base Metal Preparation

The base metal used was SS 321. The substrate was grinded to 5mm thickness and then buffed with large grain sandpaper to attain a roughness of about 50 µm. The substrates were then etched in dilute HCl solution for about an hour.

2.2 Ultrasonication Process

The Ultrasonication process was used for Graphene mixture but at low frequency. Initially, 0.05 gm of r-Go (reduced Graphene oxide) is dispersed in 20 ml of DI (Deionized Water) water and sonicated for 2 h. Then 2 gm of PVP (Poly Vinyl Pyrrolidone) is added to the sonicated mixture and sonicated at 80⁰C for 5 h with constant stirring. The system is immersed in an oil bath to maintain constant temperature.

2.3 Spin Coating

The procedure followed was very simple. A sample substrate of size 1*1 cm was placed inside the ceramic chamber of the spin coater. An earlier prepared colloidal solution of Graphene was 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 2000 rpm at room temperature. Due to the centrifugal force, the solution was evenly spread upon the substrate resulting in an even coating. This coating was then dried in a hot air oven at 80⁰C for 20 minutes.

2.4 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. In the procedure, four samples were coated using drop casting method. After this, the samples were placed in an alumina crucible and placed inside the furnace. The elevated temperature was set at 1050⁰C for about 3.30 hours. After reaching 1050⁰C, the samples were allowed to stay at that temperature for 30 minutes. Next the furnace was allowed to cool down for 24 hours before retrieving the samples.
2.5 Laser Ablation and Flame Test
The tests were carried out to determine the strength and the durability of the coating at high temperatures. Graphene-SiC coated SS 321 samples 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 out on the ablated samples. For the flame test, the samples were subjected to a gas flame which instantaneously increased the temperature for about 1 minute per sample. Finally, FESEM imaging and Surface roughness profile have been measured using SURFCOM1400.

3. Result and Discussion

As a powerful technique, SEM has been extensively used for imaging new materials, especially at micro and nano scales. Graphene consisting of a monolayer of sp2 bonded carbon atoms is a relatively new member of the carbon family.

It was observed that the Graphene surface with the coatings was smooth under all magnification. Figure 1 (a) and (b) shows the untreated and treated. The layer planes, referred to as Graphene layers, are ordered so as to be substantially parallel to one another. The bonding forces holding the Graphene layers together are only weak. Van der Waals flakes can be chemically treated to insert anion into the interlayer spacing. This shows a stable mixing of the compounds. Thus the bonding is stable. SEM images also show that the electroplated layers are fully adherent to the substrates.

![Figure 1. SEM images of graphene - SiC coated on SS 321. (a) Untreated. (b) Treated sample.](image)

![Figure 2. EDX image of. (a) Untreated. (b) Treated on coated SS 321.](image)

<p>| Table 2. EDX data of untreated SS 321 |</p>
<table>
<thead>
<tr>
<th>EL</th>
<th>AN</th>
<th>Series</th>
<th>unnn. C (wt. %)</th>
<th>norm. C (wt. %)</th>
<th>Atom. C (wt. %)</th>
<th>Error (1 sigma)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>8</td>
<td>K - series</td>
<td>38.14</td>
<td>52.02</td>
<td>73.77</td>
<td>6.50</td>
</tr>
<tr>
<td>Fe</td>
<td>26</td>
<td>K - series</td>
<td>31.85</td>
<td>43.44</td>
<td>17.65</td>
<td>2.88</td>
</tr>
<tr>
<td>C</td>
<td>6</td>
<td>K - series</td>
<td>3.33</td>
<td>4.54</td>
<td>8.58</td>
<td>1.44</td>
</tr>
</tbody>
</table>
For Surface roughness measurement the following parameters were selected, cut-off length is 4 mm and measurement speed is 0.3 mm per second. Surface roughness measurement obtained from the treated and untreated samples is shown in Figure 3 (a) and (b). The roughness values for untreated sample is 3.59 um and for treated sample is 17.2 um. This shows that the coating has been done uniformly and after the laser ablation test (treatment) there is a significance change in the surface morphology. By comparing the SEM images it is well understand that there is no crack development after the ablation test and that has been proved from the surface roughness measurement.

4. Conclusion

Upon coating with Graphene and SiC on SS 321 laser ablation and the flame test has been carried out, which shows that the coating has been done uniformly, and there are compositional changes from treated and untreated samples. From the surface roughness measurement, there is a change in the surface morphology of the treated and untreated samples, but no cracks developed due to the ablation and flame testing. Thus, Graphene and SiC coating on SS 321 increases the surface roughness and also increases the strength of the sample.

5. References

9. Zhang H, Lee HK. Plungerinneedle solidphase microextraction with graphenefla based solgel coating as sorbent for
J. Daniel Glad Stephen and G. M. Brahmanandhan


