CATALYTIC OXIDATION OF CARBON COMPOSITES

Dhanraj B1, a *, Kiran Kumar N2, b and Rama Siva Reddy K3, c ,
Dr. M. Vishnuvardhan4d, Anil Kumar V5, e

1Department of Mechanical Engineering, Vardhaman College of Engineering, Hyderabad-501218, India.
2Department of Mechanical Engineering, B.V.Raju Institute of Technology, Narsapur, Hyderabad-502313, India.
3Department of Mechanical Engineering, B.V.Raju Institute of Technology, Narsapur, Hyderabad-502313, India.
4Department of Mechanical Engineering, Vardhaman College of Engineering, Hyderabad-501218, India.
5Department of Mechanical Engineering, National Institute of Technology, Surat, Gujurat , India-

dhanraj2205@gmail.com, b kiran.nulka@bvrit.ac.in, c ramsivareddy.k@bvrit.ac.in
d mukkotivishnu@gmail.com, e veerapanenianilkumar@gmail.com.

Keywords: Aircraft brakes, catalytic oxidation, carbon composite, alkaline earth acetate

Abstract: A airbrake make use of multiple rotors for CC brakes, coupled with stators. A rotor is sourced by spokes, and the brake system provides protection for the stators. Pressing the brakes along with the hydraulic actuators performs braking action and the frictional torque is provided to stop aircrafts from overheating disc brakes. A friction between the rotating and stationary disc causes them to heat up to around 500 °C with a surface temperature of 2000 °C. He3nice, the materials used in this environment should show high thermal shock resistance. This paper focuses primarily on the study and evaluation of the effect of alkaline earth acetates on the catalytic oxidation of Carbon Composites aircraft brakes.

1. Introduction
1.1 Carbon Composite
Carbon/Carbons (CC) are a carbon matrix made up of carbon fibres. Composites of CC carbon matrix, with valuable mechanical and thermal properties at 2000 °C. CC Composites are made of carbon fibres, and almost all commercial carbon fibres are produced by turning a carbonaceous precursor into a fiber type[1]. Instead it connects the precursor fiber to make it fusible. Eventually, the cross-connected precursor fiber is heated in an inert atmosphere at a temperature of 1200 °C to 3000 °C.

The polymer Pepeg was kept in an oven for a period of 6 hours at a temperature of 700C to take away surplus solvents, subsequently a vacuum bag to compress the curing method for a period of 30 minutes[2]. It was made to 50mm cups by a water-cooled diamond saw, and nest treated by a post healing cycle in an air circulated oven at a temperature of 230°C for a period of 8 hours. Carbon composites offer the option of a heat-resistant material capable to work at temperature above 2000C percent lighter density and a low thermal expansion coefficient, this material not only survive at high temperature, when it is heated it gets more stronger[3].

Strelko et al[5] investigated on the catalytic carbon gasification originated from electron interactions among a metal and a carbon matrix ensuing in the form of carbon oxides and metal-carbon complexes. Especially, carbon dioxide formed by the thermal break down of K2CO3 activated the carbon content efficiently in making a porous structure. Saber et al[6] examined the association among K2 13CO3 and surface oxides which is carbon-bounded with temperature controlled reaction(TPR). The investigation was done and heating mixtures of 100mg of a variety of carbon molecules and 10mg of labeled K213CO3 at temperature up to 732°C in flowing helium. For raising the content of carbon the amount of CO2 released has been noted. The CO2 emitted from the K213CO3 carbon molecules was now slowly bigger than CO2 formed by carbon molecules itself. Cazorla et al [7] investigated on calcium —catalyzed carbon gasification by means of high-purity carbon that was packed with CaCO3.at 800C steady-state and step-response tests were primarily done in helium flow and then in CO2. Theoretically, it was shown that un catalyzed reaction produced
same quantity of 13CO and 12CO. the catalyzed reaction produced considerably higher concentrations of 12Co but not for increasing reaction temperature. It was also stated that a redox process relating the development of a higher oxide be supposed to the removed for the catalyzed reaction.

2. Practical setup
The experiments were conducted in the laboratory to identify the loss of carbon-carbon test samples over the specific period of duration (time), when it is subjected to conditions of oxidation simulation.

2.1 Step by step preparation
Carbon Carbon composites were impregnated with boric acid and potassium acetate and the behavior was tested in the furnace at 600 °C, 550 °C, 500 °C.

2.2 Segregation samples
DUNLOP AEROSPACE COMPANY collected small cubes of Carbon-Carbon composite samples. The samples collected from the company are used for production of aircraft brakes. Samples for observation about 30-40. the maximum and minimum weights were measured in the total sample set and were respectively 7.7436(grams) and 6.5766(grams) and average was 7.2349(grams).

![Mettler AE200 weighing machine](image)

Fig. 1. METTLERAE200 weighing machine

Maximum and minimum weights reported, and it is composed of ten samples in four sets each. Those sets are tested in Table.1

| SAMPLE SET | TEMPERATURE | CONDITION     |
|------------|-------------|---------------|
| SET1       | 600°C       | WITH IMPREGNATION |
| SET2       | 600°C       | WITH IMPREGNATION |
| SET3       | 550°C       | WITH IMPREGNATION |
| SET4       | 500°C       | WITH IMPREGNATION |
3. Impregnation of Samples

Now, the test samples were able to impregnate with solution. 15 grams of boric acid, 49.1 grams potassium acetate were taken with molar solutions 0.5 and respectively. Which dissolved into 500ml distilled water. Suppose, mixture isn’t fully dissolved into water and it should heated for the uniform solution creation. Dipped sample must be kept 40 minutes in solution. Subsequently, samples can be dried and keeping them circulation the preheated air oven, can be shown in figure.2 at 1100 °C at least for two hours and it were then removed from oven and place to cool down for 120seconds. the, the test sample are again weighed to identify the absorbed acetate. Likewise, boric acid of 5 grams and potassium acetate of 4 grams for next three sets of separate solutions. The aircraft brakes have endured various environmental conditions. Hence impregnated procedure was carried out to promote these conditions.

![Fig.2 To cool in air passing oven](image)

3.1 Heat treatment of test samples

Heat treatment of samples was done on regular period in order to know the oxidation of composite. The heat treatment of composite carried out in a called muffle furnaces . the complete process is explained in point wise

1. First taken given set of sample one and their weights are calculated
2. The heat treatment is carried out set one in a temperature 600 degree centigrade.
3. The sample set is cooled for two hours in a air circulated over for two hours
4. Again heat-treated for 600 degree centigrade after that weights are calculated to check oxidation.
5. This process is carried out for 48 hours continuous
6. The oxidation is checked with the help of weights
7. same steps are carried out for different set of samples
3.2 Microscopic Investigation

Microscopic investigation is carried out how the acetates are impregnated in the composites.

3.3 Scanning Electron Microscope (S.E.M)

After the heat treatment process, the sample tests were observed in the lens of Electron Microscope Scanning Carry JCM 5700. This test is conducted for experimental verification. The microscopic experiments were performed to learn about the structure of carbon-carbon composites, also how the composite samples had diffused the acetates and oxides. Packed with units EDS, SEM. EDS an experiment on the sample sets was carried out to micro-analyze the samples.

4. Results and Calculations:

4.1 Oxidation Test Results

Boric acid, potassium acetate were heated represents in the Table 2 for a certain amount of time at 600 °C temperature, to demonstrate the difference of the carbon – carbon samples.

| Initial Weight | Weight after Impregnation | Weight after Heat Treatment Process 2nd Hour | 3rd Hour | 4th Hour |
|----------------|---------------------------|--------------------------------------------|----------|----------|
| 6.7267         | 7.0896                    | 6.8842                                     | 6.5609   | 6.0396   |
| 6.7261         | 6.9290                    | 6.7258                                     | 6.4530   | 5.9505   |
| 6.8526         | 7.1316                    | 6.8587                                     | 6.6080   | 6.2048   |
| 6.8537         | 7.6087                    | 7.1759                                     | 6.7494   | 6.1814   |
| 7.3400         | 7.6137                    | 6.6527                                     | 6.2006   | 5.5097   |
| 7.3425         | 8.2467                    | 7.5224                                     | 6.6389   | 5.5064   |
| 7.5534         | 7.6100                    | 7.3296                                     | 6.8273   | 6.1340   |
| 7.5894         | 7.8399                    | 7.1064                                     | 6.3221   | 5.2272   |
| 7.6914         | 8.0907                    | 7.6807                                     | 7.3479   | 6.6611   |
| 7.6985         | 7.7417                    | 7.5529                                     | 7.3498   | 6.9485   |
### Table 3: Weight variation of sample set 4

| Initial weight | 2\(^{nd}\) hour | Weight loss | 3\(^{rd}\) hour | Weight loss | 4\(^{th}\) hour | Weight loss |
|----------------|------------------|-------------|------------------|-------------|-----------------|-------------|
| 6.7257         | 6.8842           | -0.158      | 6.5609           | 0.3233      | 6.0396          | -0.521      |
| 6.7261         | 6.7258           | 0.0003      | 6.453            | 0.2758      | 5.9505          | 0.5025      |
| 6.6526         | 6.8587           | -0.006      | 6.608            | 0.2507      | 6.2048          | 0.4032      |
| 6.8537         | 7.1759           | -0.322      | 6.7494           | 0.4265      | 6.1814          | 0.568       |
| 7.34           | 6.6527           | 0.6873      | 6.2006           | 0.4521      | 5.5097          | 0.6909      |
| 7.3425         | 7.5224           | -0.179      | 6.6389           | 0.8835      | 5.5064          | 1.1325      |
| 7.5534         | 7.2396           | 0.3138      | 6.8273           | 0.4123      | 6.134           | 0.6933      |
| 7.5894         | 7.1064           | 0.463       | 6.3221           | 0.7843      | 5.2272          | 1.0949      |
| 7.6914         | 7.6807           | 0.0107      | 7.3479           | 0.3328      | 6.6611          | 0.6868      |
| 7.6985         | 7.5529           | 0.1456      | 7.3498           | 0.2031      | 6.9484          | 0.4014      |

### Table 4: Weight variation of potassium acetate at 600°C

| Initial Weight | Weight after Impregnation | Weight after Heat Treatment |
|----------------|---------------------------|----------------------------|
|                | 2\(^{nd}\) Hour | 3\(^{rd}\) Hour | 4\(^{th}\) Hour |
| 7.2161         | 7.3271   | 4.3642    | 2.9054    | 1.6691    |
| 7.2206         | 7.3672   | 4.7269    | 3.2123    | 2.0154    |
| 7.2412         | 7.4239   | 4.2466    | 3.6786    | 2.973     |
| 7.43           | 7.5922   | 4.5864    | 3.5342    | 2.4887    |
| 7.7328         | 7.8780   | 4.1974    | 3.4426    | 2.7628    |

### Table 5: Weight deficiency of potassium acetate at 600°C

| Initial weight | 2\(^{nd}\) hour | 3\(^{rd}\) hour | 4\(^{th}\) hour |
|----------------|-----------------|-----------------|-----------------|
|                | Weight loss     | Weight loss     | Weight loss     |
| 7.2161         | 4.36442         | 4.7269          | 4.2466          | 4.5864      | 4.1974      |
| 7.2206         | 2.8519          | 2.4937          | 2.9946          | 2.8436      | 3.5354      |
| 7.2412         | 2.9054          | 3.2123          | 3.6786          | 3.5342      | 3.4426      |
| 7.43           | 1.4588          | 1.5146          | 0.568           | 1.0522      | 0.7538      |
| 7.7328         | 1.6691          | 2.014           | 2.973           | 2.4887      | 2.7628      |
|                | 1.2363          | 1.1969          | 0.7056          | 1.0455      | 0.6798      |
5. Analysis of results:
The below figure displays the rate of percentage carbon losses to the specific time for potassium acetates, boric oxide, potassium acetates and boric acid, respectively.

![Graph of Time vs % avg Carbon Loss](image1)

**Fig. 4 C vs. sample set2 at 600°C**

![Graph of Time vs % avg Carbon Loss](image2)

**Fig. 5 C vs. ko2 a at 600°C**

y = 13.868x - 4.9029

**Linear (Time Vs %avg Carbon Loss)**
5.1 Activation Energy Calculation

In Activation Energy shown below, the energy needed to resolve the before reactants can be converted to products. It may take very quick, if activation energy is at low temperature.

![Activation Energy](image)

**Fig.6 Activation of Energy**

5.2 Reaction rate governed by Arrhenius equation

Energy of activation can be determined by intrigueIn (k) with the graphs of reciprocal temperature. The rate constant derived from slope, and the % of carbon losses over the time. In figure below, the graph obtained by combining boric , potassium acetate, boric acid and potassium acetate shall be given.

![Arrhenius test samples](image)

**Fig.7 Arrhenius test samples- impregnation**

The slope is of $-\frac{Q}{R}$, and on each Arrhenius plot was superimposed on a best-fit line for getting value for the slope.

Slope=$-\frac{Q}{R}$, Where $Q$=Energy Activation, $R$= gas constant

$-5284.3=-\frac{Q}{8.391}$

Therefore, $Q=42469.72$J/mole

For mixture of potassium acetate & boric oxide the activation energy is required to resolve reactions is 42469.72J/mole.
6. EDS Analysis

Under EDS analysis, the samples were checked to check whether in the c-c composites Where Diffusion of Acetates. The JCM 5700 SEM has an EDS to it. And the figure represents the different elements and how much of carbon-carbon composites samples are available, C-C samples also contained potassium, silicon, oxygen, and boron as shown in the graph below.

![Fig.8 Visualization of CC Composites in EDS Analysis](image)

6.1 EDS -Spectra (Observation)- CC Composite

The below figure represents the boric oxide on sample surface blocks oxygen access to fiber.

![Fig.9 boric oxide in white color](image)

7. Conclusions:

Similar to the boric acid, potassium acetate and potassium acetate alone, the catalytic effect of the potassium acetate and boric oxide mixture has stronger results from the experimental tests. Combined potassium acetate and boric oxide exhibit identical properties on C-C test samples as validate sodium acetate. And from the above graphs, it can represent that, on c-c samples the amount of carbon loss over time has more use. It has been confirmed from previous studies the boron oxide serves as an inhibits throughout oxidation reaction.

Energy activation, for potassium acetate and boric oxide a is $Q=42469.72$ J/mole, means the reactants required to transform products to reactions are very less compared with boric oxide ($Q=57390.9426$ J / Mole) mixtures the magnesium and potassium acetate ($Q=51587.772$ J /mole) Potassium acetates needed less quantities of triggering energy which is much lesser than combined potassium acetate and boric acid to turn reactants to the products. It showed weaker
catalytic effect of potassium acetate on C-C Composite test samples. Oxidation. Although, it shows extraordinary non-corrosive properties.

And the potassium acetate and boric acid can be considered to serve as an inhibitor throughout oxidation reaction. Potassium acetate are good catalyst for sample oxidation and it couldn’t be used in the practical defense against oxidation.

Thus, potassium acetate and boric acid efficient catalysis for the oxidation of c-c samples and therefore not a practical defense against oxidation. But, several other alternative means of protection are used by the aircraft industry instead of using boric oxide, potassium-acetate respectively.

Future studies on C-C tests of the mixture of boric oxide and potassium acetate, potassium acetate and magnesium acetate and potassium acetate and calcium acetate will be carried out. Compared with the present experiment conducted, that could provide better results.

8. References
[1] Wu and Radovic, catalytic oxidation of carbon/carbon composite materials in the presence of potassium and calcium acetates, 2005, pp. 333-344.
[2] Wu and Radovic inhibition of catalytic oxidation of carbon/carbon composites by boron doping, 2005, pp. 1768-1777.
[3] David A.Fox and Alfred H.White Effect of Sodium Carbonate upon Gasification of Carbon and Production of Producer Gas, 2002, pp.259-266.
[4] JA Moulijn, F Kapteijn, Towards a unified theory of reactions of carbon with oxygen containing Molecules, 1995, pp.1155-1165.
[5] V.V. Strelko, N.V. Gerasimenko, N.T. Kartel’, Low-temperature activation and oxidation of carbon Materials impregnated with potassium carbonate, Russ. J. pp. 286-289.
[6] J.M. Saber, J.L. Falconer, L.F. Brown, Interaction of potassium carbonate with surface oxides of carbon, 1968, pp.1356-1359.
[7] D. Cazorla-Amoros, A. Linares-Solano, F.H.M. Dekker, F. Kapteijn, Isotopic steady-state and step response study on carbon gasification catalyzed by calcium, 1995, pp. 1147-1154.
[8] H Zoheidi, DJ Miller, Role of oxygen surface groups in catalysis of hydrogasification of carbon black by potassium carbonate, 1987, pp. 809-819.
[9] Ushakiran Kumar sanivada MSc major project 2008/2009 (university of Hertfordshire) [10]Levar Johnson final year project 2006/2007 (university of Hertfordshire)
[10] http://www.so.soton.ac.uk/staff/pmrp/GY102/module1/m1f9.GIF
[11] http://www.chemguide.co.uk/physical/basicrates/arrhenius1.gif