**CO2 Plasma Arc Heating Test in C/C Composite Penetrated with Silicone Oil**

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In an attempt to study the oxidation phenomena of the thermal protective materials, test on carbon fiber reinforced carbon (C/C) composite have been conducted in plasma arc facility under the similarly MARS and other planet entry conditions. An arc heated wind tunnel used for thermal protective testing of ablation materials for capsule, probe and space vehicle. The study of attempts of ablation phenomena related the plasma characteristics to the thermally decomposed gas, the chemical composition and material structure after an arc heating test. Sample heating test during virgin C/C and C/C composite penetrated with dimethyl silicone oil target in a CO2 arcjet flow is experimentally investigated on measurements of emission spectra in shock layer and internal temperature distribution in the ablation materials.

**Key Words:** High Enthalpy Flow, Plasma Arc, Ablation, Spectroscopic Measurement

### Nomenclature

\[d_e\] : exit diameter [mm]  
\[d_t\] : throat diameter [mm]  
\[M\] : Mach number [-]  
\[T\] : temperature [K]  
\[t\] : elapsed time [s]

### Subscripts

\[r\] : rotational  
\[v\] : vibrational

### 1. Introduction

Arc heated wind tunnel produce high enthalpy and high speed flow which are used to research heating and ablating phenomena in reentry flow for thermal protective materials(Phenolic Impregnated Carbon Ablator (PICA), carbon fiber reinforced carbon (C/C) composite, SiC, etc). Arc heater was used in these tests to predict the material heat capacity for the design of space probe. The supersonic experiments showed spectroscopic profile, temperatures in shock layer and inspection of the material structure under the before heating and after each cycle\(^1\)\(^-\)\(^3\). The study of kinetics and attempts of ablating phenomena related the plasma characteristics to the thermally decomposed gas, the chemical composition and structure. In the present work, time resolved temperature distribution and the spectroscopic measurements of plasma arc exposed by arc heating test during the C/C composite target in a CO2 arcjet flow is studied experimentally.

### 2. Experiments\(^7\)

The high enthalpy and high temperature gas flow can be constructed for several hollow type arc heated wind tunnel, such as the segmented constrictor type, Hüls type and Hybrid type. In this study, supersonic tests were conducted by using a Hüls type DC arc heated wind tunnel facility.

#### 2.1. Hüls type arc heater

Figures 1 and 2 show the description of a Hüls(Huels) type arc heated wind tunnel. The facility was constructed an arc heater, hollow electrode for cathode and anode, test chamber, low density tank and DC power supply. The heater connected to the supersonic nozzle, test chamber and low density tank with 2.05m\(^3\) in volume. Both electrodes and the supersonic nozzle were independently cooled by tap water. The electrodes were made of oxygen free copper. The inner diameters of the cathode and anode have 12 and 18mm, respectively. The conical supersonic nozzle is 3.5mm in throat diameter \(d_t\) and 10.5mm in exit diameter \(d_e\), therefore designed Mach number \(M\) is 3.0 include the 30% of boundary layer thickness. The unit consists of two coaxial electrodes separated by an insulator(Teflon) in the plenum chamber. Test gases used for carbon dioxide, were injected tangentially into the plenum chamber to make swirl flow. To avoid any undesirable concentrated current spot of discharge at some fixed position of the heating tube surface, a magnetic coil system is attached to the cathode side of arc heater coaxially to the tube axis. The pressure in low density tank and the plenum chamber were kept at 13.3Pa(0.1torr) and 0.1213MPa, respectively. The discharge current, discharge voltage and mass flow tare were tested at 180A, 90V and 0.78g/s, respectively. At the ablation...
testing under the CO2 plasma arc flow, a circular cylinder model is placed 21mm downstream of the supersonic nozzle exit for 180s of arc running. Time history of internal temperature in thermal protective material was measured by K type thermocouple at position of 10mm from the front surface of materials.

**2.2. Spectroscopic measurement apparatus**

The emission spectra were spectroscopically measured in the shock layer. Figure 3 shows the schematically description of the spectroscopic measurement system. The spectra profiles were measured by high resolution spectrometer (SOLAR TII Inc, MS7504) with high response ICCD (Andor Inc, i-Star DH740-18F-03) for CO2 arc plasma flow. The spectrometer was operated 750mm focal length with 2400 grooves/mm, 500nm blaze ruled grating and an entrance slit width of 0.03mm. This measurement system allows a spectral resolution of 0.0055nm, and scanning duration is of 0.5s. Spectral and spatial resolutions are given simultaneously allowing a good accuracy.

**2.3. Thermal protective material**

In ablation testing, C/C composite (AC200) were manufactured by Across Co., Japan. The physical characteristics of C/C composite are given in Table 1. Thermal protective material was about 10mm in diameter and 40mm in length. C/C composite made from carbon fiber reinforced carbon matrix. The carbon fiber orientations have (0/90) cross-ply lamination layers. It has high specific strength comparison with other metal materials, and withstands temperature over 1700K.

| Physical characteristics of C/C composite(AC200). |
|---------------------------------|-----------------|
| Density [kg/m³]                 | 1700            |
| Heat treatment [K]              | 1700            |
| Carbon fiber content [%]        | 40              |
| Mass [g]                        | 2.479           |

Vacuum pressure impregnation (VPI) is possible remove air, any gas and moisture from the material. Pressure was hold at vacuum (4kPa) and impregnated silicone oil in the material inside for 24H. The oil is quantity of impregnation from 7 to 9% of dry mass in C/C composite. Dimethyl silicone oil (KF-96-10CS) is produced by Shin-Etsu Chemical Co., Ltd, as shown in Table 2. Figure 4 shows the molecular structure of dimethyl silicone oil. The dimethyl group containing one carbon atom bonded to three hydrogen atoms CH₃.

| Comparison of physical properties of silicone oil. |
|---------------------------------|-----------------|
| Density [kg/m³]                | 935.0           |
| Kinetic viscosity [mm²/s]      | 10.0            |
| Specific heat [J/kg·K]         | 140.0           |
| Thermal conductivity [W/m·K]   | 11.7×10⁻³       |

**3. Results and Discussion**

Figures 5a and 5b show the time dependent of the arc heating supersonic test in virgin C/C and oil penetration C/C, respectively. Frame 1 is starting an arc heating. Frame 2 to 7 and frame 7 to 12 is recorded every 5 and 30s, respectively. In Figs. 5a and 5b, the each material is gradually heating by arc jet flow at front region. Therefore, they observed that a bow shock occurs in front of the body. After time \( t = 10 \)s, the each materials is gradually heating by an arcjet flow at front region. The material is melting and abraded by the arc heating. For frames 1-5 in Fig. 5b, the vaporization of silicone oil spouts from the material, and after evaporation, the oxidation film of SiO2 attaches to material surface. The amount of heat it absorbs from arc flow. Figures 6a and 6b show comparisons of
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virgin C/C at a and oil penetration C/C at b after \( t=180 \text{s} \) of arc heating. Thickness losses are about 5.4mm in Fig. 6a and about 2.5mm in Fig. 6b for the present arc heating conditions. In the case of virgin C/C, material structure after the arcjet testing was carbonized to be a char layer and melting due to dissociate CO2 plasma. While oil penetration C/C, catalytic of C/C material made a SiO2 that it is predicted to oxidation and recombination of atom O.

Figure 7 show typical emission spectra profiles at shock layer for both materials under CO2 plasma arc heating test. The identified transitions are attributed to C2 SWAN molecular bands, atomic lines C, O and Cu. From experimental measurements, molecular band of SWAN system is very intense in the shock layer due to the homo-nuclear having dipole moment. In Fig. 7, the intense CII and OI series emission lines are at 426.7nm \((2s^23d \rightarrow 2s^22p^3p)\), 589.16nm \((2s^23d \rightarrow 2s^22p)\) and from 551.2 to 551.3nm \((2s^22p^63p \rightarrow 2s^22p^66d)\), respectively.

![Figure 5](image5.png)

(a) Virgin C/C
(b) Oil penetration C/C

Fig. 5. Ablation testing of C/C composite under the CO2 plasma arc flow.

![Figure 6](image6.png)

(a) Virgin C/C
(b) Oil penetration C/C

Fig. 6. Photographs of ablation test in C/C composite under the arc heated carbon dioxide flow.

![Figure 7](image7.png)

Fig. 7. Measured emission spectra in the shock layer of both test piece under CO2 arc jet flow.

Many emission lines for atom O are indicated to oxidation and recombination for C/C composite penetrated with silicone oil. Therefore, the oxidation of oil penetration C/C is stronger than virgin C/C.

Figures 8a, 8b, 9a and 9b show the Boltzmann plot of vibrational and rotational level at shock layer. Vibrational and rotational temperatures in C2 SWAN band are determined from Boltzmann plot method by using the ratio of band heads from different vibrational and rotational levels. The band head inclined for \((0,0)-(2,2)\) of the \(d^3\Pi_u-a^3\Pi_g\) transition of C2 SWAN band. Boltzmann plot of rotational level \((37<J<47)\) in shock layer. Experimental vibrational and rotational temperature for virgin C/C and Oil penetration C/C were calculated to be about 2700K \(\pm 200\)K, 2800K \(\pm 300\)K, 3100K \(\pm 700\)K and 3200K \(\pm 400\)K, respectively. The rotational temperatures are lower than vibrational temperatures, which show the shock layer becomes the thermal non-equilibrium condition.

Figure 10 shows time histories of black-body temperature of virgin C/C and oil penetration C/C. Black-body temperature increases as emission spectra intensity increase by arc heating time. Black-body temperature of oil penetration C/C is higher than virgin C/C condition due to deposition of SiO2.
Figure 11 shows time histories of internal temperature of virgin C/C and oil penetration C/C at position of 10mm from the front surface of materials. Internal temperatures arise with arc heating time by high enthalpy CO2 flow. Then CO2 arcjet flow induced material surface that a bow shock occurs at front face of the body. After time $t=50$s, the internal temperature in the each materials became slow comparison with 0-50s. Maximum internal temperature for both materials was measured to be 1170 and 1090K, respectively. The material is abraded by the arc heated flow. Sample heating test was finished at 180s, temperature decreased quasi-exponentially until room temperature. Internal temperature gradient ($\Delta \text{Temp}/\Delta \text{time}, \text{K/s}$) were evaluated from Figure 11. $\Delta \text{Temp}/\Delta \text{time}$ were calculated to be about 24.2K/s in virgin C/C and about 11.3K/s in oil penetration C/C. From the results, thermal protection temperature of oil penetration C/C rises by deposition of SiO2. Effect of thermal protection can improve by penetrating with silicone oil.

4. Conclusions

This research is to study supersonic heating test in thermal protective materials, and spectra profile in shock layer and temperature measurement under CO2 arc plasma flow.

During the arc heating, the vaporization of silicone oil is spouting from the material. After the evaporation, the oxidation film of SiO2 attaches on material surface.

Material structure of virgin C/C after the arc heating is
carbonized to be a char layer due to high temperature CO2 ambiance, while oil penetration made a SiO2 on its surface that it is predicted to oxidation and recombination of atom O.

Mass loss in virgin C/C is larger than oil penetration C/C, which shows ablation gas is emitting in shock layer. Vibrational-rotational emission spectra profiles can be measured using a high resolution and high response spectroscopic measurement system. The spectral profiles in the shock layer of oil penetration C/C was measured C2 SWAN molecular bands, atomic lines C and O due to the ejection of ablation gas from the material. Experimental vibrational and rotational temperatures were evaluated by Boltzmann plot from C2 SWAN molecular bands at about 2700K±300K, 3100K±700K for virgin C/C and 2800K±300K, 3200K±400K for oil penetration C/C, respectively. The vibrational and rotational temperatures in oil penetration C/C were larger than virgin C/C, and gas temperature increases by Si precipitated from silicone oil by a hot gas. Internal temperature gradients were measured to be about 24.2K/s (ΔTemp/Δtime) in virgin C/C and about 11.3K/s in oil penetration C/C. Internal temperature gradient in oil penetration C/C has half value of virgin C/C, whereas thermal protection improves by penetrating with silicone oil. It is seen from this results show that oil penetration material has good property of thermal protection under the CO2 plasma arc.

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References


