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## EXPERIMENTAL ASSESSMENT OF FIBER REINFORCED CERAMICS FOR COMBUSTOR WALLS



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### ABSTRACT

Experimental and theoretical work concerning the application of ceramic components in small high temperature gas turbines has been performed for several years. The significance of some non-oxide ceramic materials for gas turbines in particular is based on their excellent high temperature properties. The application of ceramic materials allows an increase of the turbine inlet temperature resulting in higher efficiencies and a reduction of pollution emissions.

The inherent brittleness of monolithic ceramic materials can be virtually reduced by reinforcement with ceramic fibers leading to a quasi-ductile behavior. Unfortunately, some problems arise due to oxidation of these composite materials in the presence of hot gas flow containing oxygen.

At the Motoren- und Turbinen Union, München GmbH, comprehensive investigations including strength, oxidation, and thermal shock tests of several materials that seemed to be appropriate for combustor liner applications were undertaken. As a result, C/C, SiC/SiC, and two C/SiC-composites coated with SiC, as oxidation protection, were chosen for examination in a gas turbine combustion chamber.

To prove the suitability of these materials under real engine conditions, the fiber reinforced flame tubes were installed in a small gas turbine operating under varying conditions. The loading of the flame tubes was characterized by wall temperature measurements.

The materials showed different oxidation behavior when exposed to the hot gas flow. Inspection of the C/SiC-composites revealed debonding of the coatings. The C/C- and

the SiC/SiC-materials withstood the tests with a maximum cumulated test duration of 90 hours without damage.

### INTRODUCTION

Gas turbine development today concentrates on higher thermal efficiencies and lower pollution emissions. One possible approach is an increase of turbine inlet temperature. Therefore, either an effective cooling of the components forming the hot-section or high temperature resistant materials are required. Carbon (C) or silicon carbide (SiC) based materials with their outstanding high temperature properties, offer a great potential for such applications (Strife and Sheehan, [1988]; Lamouroux and Camus, [1994]).

However, the advantageous qualities of monolithic materials in structural applications, such as their high Young's modulus, high thermal conductivity, and excellent oxidation resistance, are partly offset by their relatively low fracture toughness. A wide range of additives and particulate reinforcements have been tried as toughening agents. Reinforcement with SiC or C continuous fibers could yield the highest fracture toughness. These materials show an increase in fracture toughness by one order of magnitude and an increase in fracture energy by two orders of magnitude (Helmer et al., [1995]). Since they have to operate under hostile stress-temperature environment conditions, the choice of toughening phases is restricted by high temperature chemical incompatibility and by thermal expansion coefficient mismatch with the matrix.

In addition, the desired applications involve extended periods of operating in oxidizing atmospheres which require a proven oxidation resistance of the composites (Kirk, [1989]).

Therefore, at the Institut für Thermische Strömungsmaschinen (ITS), in cooperation with the Motoren- und Turbinen Union München GmbH, different SiC and C based composite materials were tested for their oxidation behavior. In preliminary investigations, the materials exposed to high temperatures in an atmosphere containing oxygen were judged by mass reduction and strength tests. The most promising materials were selected for manufacturing flame tubes to test their durability under real engine conditions. The new flame tubes were tested in the ceramic combustion chamber of the Klöckner Humboldt Deutz T216 type gas turbine that was designed at the ITS (Münz et al., [1996]).

## MATERIALS SELECTION

Three different composite materials were chosen for the tests under real engine conditions. These materials were selected out of seven SiC or C composites and four fiber reinforced glasses. They offered the most promising high temperature properties which are necessary for combustor walls. For example the glass materials suffered from a drastic strength loss for temperatures exceeding 1000°C (Andrees et al., [1996]). For reasons of brevity, in the following section only the preliminary test results of the materials selected are shown (see Table 1).

| Composite | Interface coating | Fabrice (layup)        | Manufacturer       |
|-----------|-------------------|------------------------|--------------------|
| C/C       | -                 | satin weave (2D-0/90°) | Schunk             |
| C/SiC     | PyC               | UD laminate (2D-0/90°) | Domier             |
| SiC/SiC   | PyC               | plain weave (2D-0/90°) | MAN Technologie AG |

Table 1: Selected fiber reinforced materials.

Under oxidizing atmospheres, fiber degradation was expected in the two-directional composites reinforced with silicon carbide fibers. In the case of the materials with carbon fibers, rapid oxidation was expected. The mass reduction of the composites reflects the structural changes of the specimens. The qualification was done by strength measurements with respect to the mass loss. The experiments were done in a muffle furnace at elevated temperatures. Figure 1 shows the mass reduction of the carbon reinforced materials versus time for two different temperatures, and in Figure 2 the strength dependency on mass reduction is demonstrated. Hence, a life expectancy can be determined if the lowest allowable strength is prescribed.

Assuming a minimum tensile strength of 150 MPa is sufficient for reliable operation of the component, the durability in a 1000°C atmosphere would be only 8 minutes

for the C/C material and only 6 minutes for the C/SiC material. This clearly shows the need for effective external oxidation protection.

Since coatings can influence the mechanical properties of the base material, strength tests were conducted for coated specimens. It was demonstrated that SiC-coated C/C and C/SiC showed reduced tensile strengths depending on the coating process. This is a result of the brittle surface and an increase in the cross sectional area of the specimen caused by the coating that has poor mechanical properties. However, the protected materials showed a significant reduction in mass loss while maintaining comparatively high tensile strengths, when exposed to the hot gas atmosphere (see Table 2).

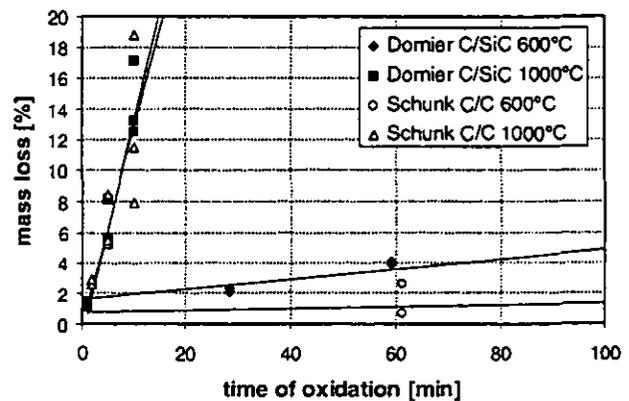


Figure 1: Mass loss versus oxidation time for composite materials.

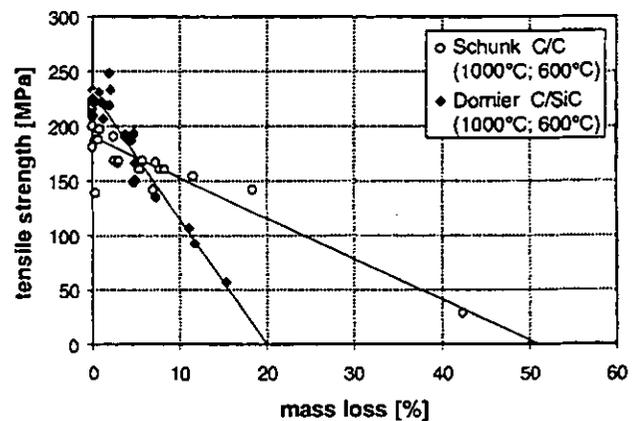


Figure 2: Strength dependence on mass loss.

Since the carbon reinforced composites did not show sufficient oxidation resistance, a SiC/SiC material with a SiC and an additional glaze-based silicate ( $\text{SiO}_2$ ) coating was added to the test program. The oxidation test results are also listed in Table 2.

Comparing the C and SiC reinforced materials, the superiority of the SiC/SiC can be clearly seen. The reason for

this superiority is that the degradation of the SiC fibers seems to be not as critical as the oxidation of the carbon fibers.

Out of these materials four flame tubes were manufactured for tests under engine conditions (see Table 3). The fiber architecture and the interface coating correspond to the tested specimens. The carbon reinforced composites were coated with SiC either by chemical vapor deposition (CVD) or a dross process. The SiC/SiC material was protected by a CVD-SiC layer.

|                | Composite | Mass loss [%] | Tensile strength [MPa] |
|----------------|-----------|---------------|------------------------|
| as delivered   | C/C       | -             | 90-93                  |
| 1230°C/ 1000 h | C/C       | 8.1-8.6       | 85-99                  |
| 1430°C/ 100 h  | C/C       | 7.8-8.4       | 101-130                |
| as delivered   | C/SiC     | -             | 198-252                |
| 1230°C/ 1000 h | C/SiC     | 7.5-25.5      | destroyed              |
| 1430°C/ 100 h  | C/SiC     | 8.5-18.9      | 77-96                  |
| as delivered   | SiC/SiC   | -             | 284.7                  |
| 1230°C/ 1000 h | SiC/SiC   | -             | 117-130                |
| 1300°C/ 100 h  | SiC/SiC   | 0.22-0.37     | 140-156                |

Table 2: Oxidation behavior of coated composites.

It has to be mentioned that the protected C/C composite for the engine tests does not conform with the material listed in Table 2, since this material suffered from poor mechanical properties. According to Schunk Company the flame tube material exhibits a much better strength due to a modified CVD process for the coating of the base material.

| Composite | Oxidation protection | Manufacturer       |
|-----------|----------------------|--------------------|
| C/C       | CVD-SiC              | Schunk             |
| C/SiC     | CVD-SiC              | Dornier            |
| C/SiC     | dross SiC            | Dornier            |
| SiC/SiC   | CVD-SiC              | MAN Technologie AG |

Table 3: Flame tube materials.

## COMBUSTION CHAMBER

A cross sectional view of the ceramic can-type combustor is given in Figure 3. The combustor walls consist of three layers. The inner layer is a hot gas resistant composite, the middle layer is a flexible oxide fiber material, and the outer layer is a metal casing. The flexible insulation in the radial direction, and the spring support of the swirler in the axial direction allows for an almost unhindered thermal expansion of the ceramic flame tube. This reduces the critical tensile stresses to a minimum, especially when considering the

thermal expansion mismatch of the metal containment and the composite liner wall. The insulation keeps the fiber reinforced material at a more homogeneous temperature distribution and lowers the thermal stress level. The temperature loading of the combustor liner is measured by thermocouples along the outer ceramic wall. Flanges on the outer side of the combustion chamber give access to the flame tube for measuring the radial temperature distribution with transversable PtRh-Pt thermocouples (not shown in Figure 3). The measuring planes are declined by 3° to detect the streaks of the cold dilution air.

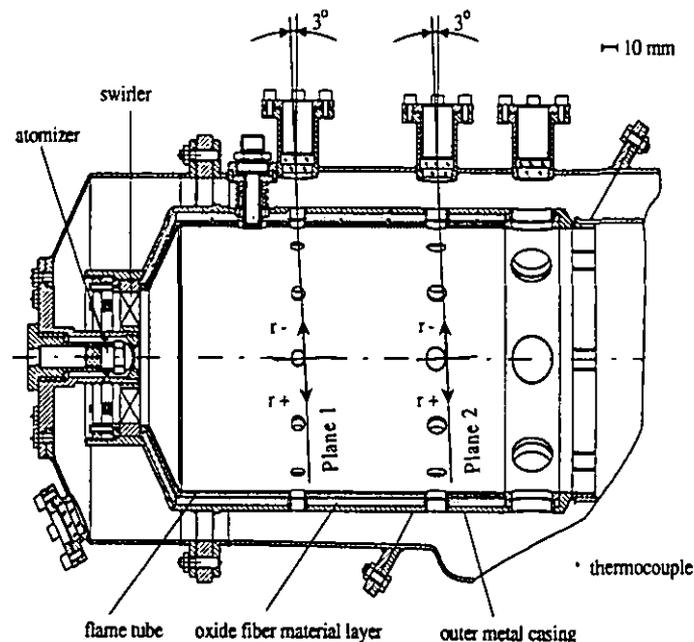


Figure 3: Flame tube construction.

This newly designed ceramic combustion chamber was integrated into a Klöckner Humboldt Deutz T216 type gas turbine. The output power of the engine is 74 kW at 50,000 rpm. The nominal pressure ratio is 2.8 and the air mass flow is 0.9 kg/s at a turbine inlet temperature of 810°C.

The dome of the combustor consists of sintered silicon carbide. Since large holes for the dilution air are necessary at the end of the flame tube, this part is separated from the ceramic flame tube and made of a Nickel alloy. The flame tube itself has a length of 210 mm and an inner diameter of 144 mm. The thickness of the ceramic wall is 3 mm. The fiber reinforced flame tubes were made by the manufacturers indicated in Table 3. The pipe specimens were installed into the combustion chamber and exposed to the hot gas flow under real engine conditions. Figure 4 shows a photograph of the flame tube assembled with the SiC/SiC tube from MAN Technology.

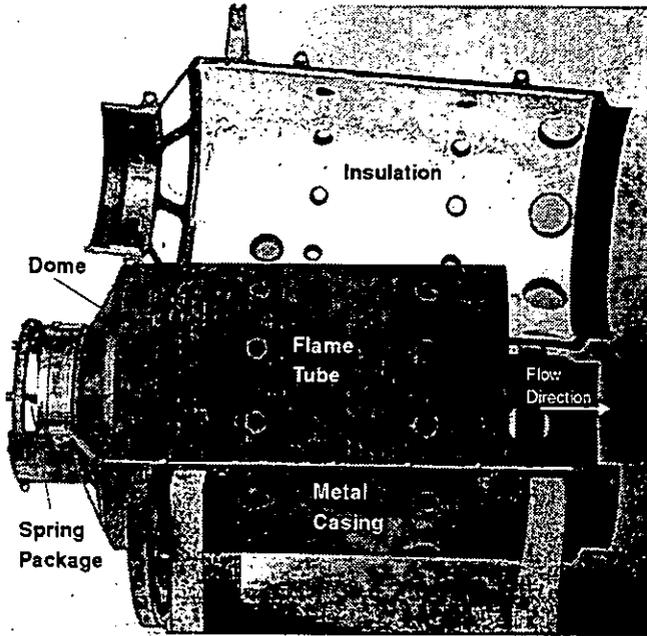


Figure 4: Ceramic flame tube.

## EXPERIMENTAL RESULTS

During machine operation, all characteristic parameters such as pressures, temperatures, speed, power etc. were recorded. The wall temperatures were used to determine the thermal loading of the composite materials. Figure 5 displays typical axial temperature distributions of the ceramic wall. The lines refer to different operating conditions. As expected, the thermal loading increases with higher rotational speeds. Increasing the output power also causes higher temperature loading, especially near the exit of the flame tube. This is due to the elongation of the flame under these conditions.

The gas turbine was run under various operating points. The highest outer wall temperature observed was about 1050°C and was detected between the dilution holes in the middle of the flame tubes at nominal speed. The temperature distributions were similar for all flame tubes tested.

The accumulated testing time for the carbon reinforced composites was limited to 10 hours. At first, operating points referring to lower thermal loads were tested. With increasing test duration the thermal load was intensified. Between the single tests the flame tubes were inspected visually and morphological changes were documented by macro photography. Hot-gas profiles were measured for all operating conditions. An example of the radial temperature distribution is plotted in Figure 6. This graph shows that for nominal speed the flame establishes right between the measurement planes. The influence of the injected air can be seen at relatively cold zones near the wall.

All four flame tubes withstood the thermal load under an oxidizing atmosphere without severe damage. The Dornier flame tube, coated with the dross SiC layer, showed debonding on the inner surface at the highest load section. Since preliminary studies revealed that this material does not have a long life expectancy without oxidation protection, this coating and composite combination can not guarantee reliable long term machine operation (see Figure 7).

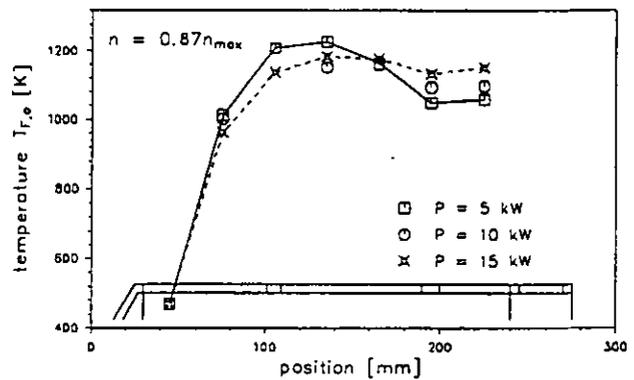
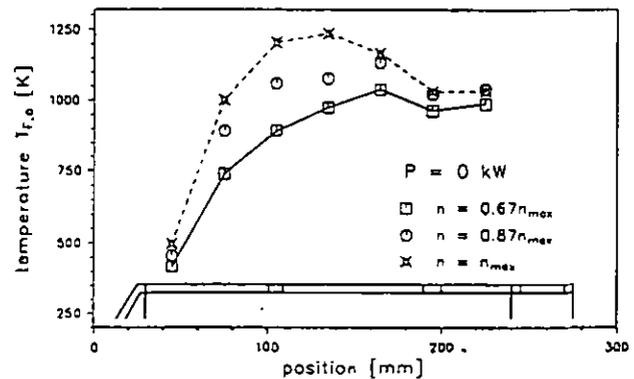


Figure 5: Temperature distributions on the outer surface of the flame tube.

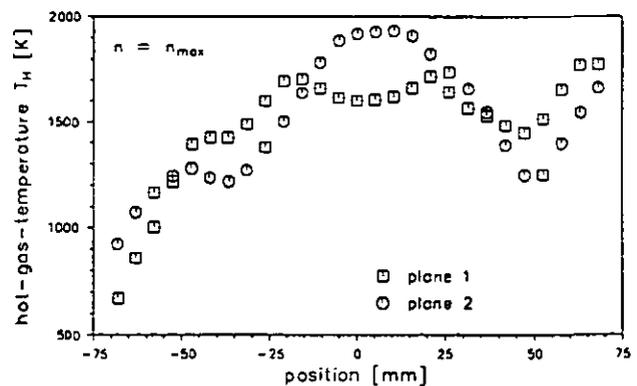


Figure 6: Radial hot-gas temperature distribution in the flame tube.

The second Dornier flame tube coated with CVD-SiC, showed no damage on the inner side. However, on the outer side a small part of the coat chipped off. This is believed to be a result of a mismatch between the thermal expansion coefficients. The tube was still in good condition, after operating for 10 hours, and did not exhibit further damage. The thermally high-loaded regions showed an intensive discoloring. This phenomenon was detected on all flame tubes except the one coated with dross SiC. The discolorations are due to establishing a  $\text{SiO}_2$  layer which results from the oxidation process and helps to suppress the subsequent oxidation. The thin amorphous glass layers reflect the different wavelengths of the incoming light depending on their thickness. As a result, the surface appears with rainbowlike colors (Figure 8).

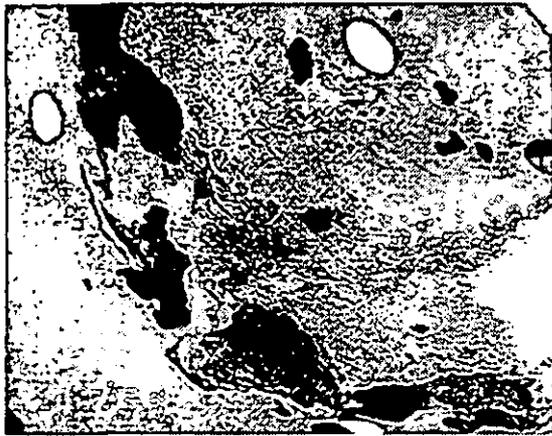


Figure 7: C/SiC flame tube with dross SiC coat after 10 hours of engine operation.

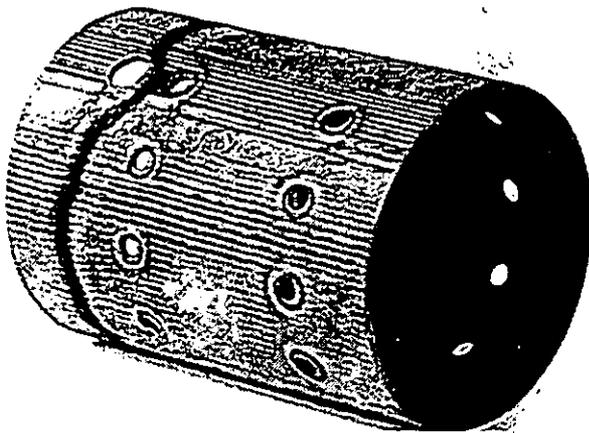


Figure 8: C/SiC flame tube with CVD-SiC coat after 10 hours of engine operation.

The C/C flame tube that was manufactured by Schunk withstood testing with no structural defects. Only the surface color had changed after 10 hours of operation (Figure 9).

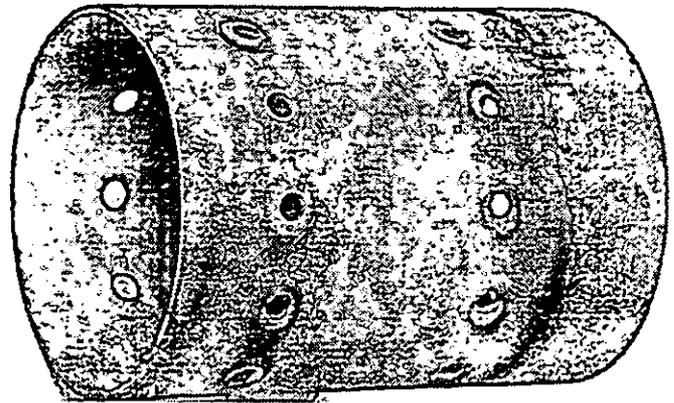


Figure 9: C/C flame tube with CVD-SiC coat after 10 hours of engine operation.

The test program was extended for the promising SiC/SiC composite. Besides the alteration in surface color, no significant change was detected after 10 hours. The only significant difference to the "as delivered" state, was a small area of debonding at the front face of the pipe. This was due to chemical reactions between the Ni-based metallic ring element and the ceramic composite material. The manufacturer, MAN Technology AG, observed similar reactions in other applications. Therefore, in the following tests, the SiC composite was separated from the metal by a thin interface layer which consisted of a fiber oxide material.



Figure 10: Inner side of the SiC/SiC flame tube after 30 hours of engine operation.

The SiC/SiC combustion chamber, in operation now for more than 90 hours, exhibited no damage. It also had to withstand numerous start and shut down processes which caused critical loads because of the high temperature

gradients. Downstream of the dilution holes, the surface was colored brown. This was probably a result of deposits from the incoming air. In Figure 10 the area downstream of a hole in the flame tube is shown. The position of soot deposits also demonstrate the tangential velocity component of the swirling air flow in the combustion chamber.

The tests under real engine conditions seem to confirm the superior properties of this silicon carbide reinforced composite. The material withstands the oxidizing atmosphere with no structural change, as can be seen in Figure 11.

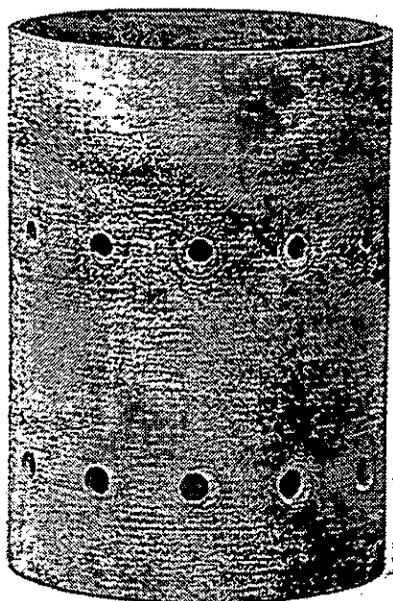


Figure 11: SiC/SiC flame tube after 50 hours of engine operation.

## CONCLUSIONS

Four flame tubes made of composite materials with a variety of coatings for oxidation protection were tested under real turbine conditions. Preliminary studies by the Motoren und Turbinen Union, München GmbH, helped in identifying the most promising composites. The flame tubes were installed into the ceramic combustion chamber developed at the Institut für Thermische Strömungsmaschinen.

First examinations of the uncoated composites showed that oxidation of the C-fibers was a very rapid process, and it was concluded that oxidation protection is necessary. Tests with coated specimens in temperature regimes between 1200°C and 1400°C revealed that the degradation of the SiC-fibers is less critical. As a result, the SiC/SiC material showed the best oxidation resistance.

After operating 10 hours in the Klöckner Humboldt Deutz T216 type gas turbine, both C/SiC flame tubes showed debonding of the different coatings. This debonding was probably due to the differing thermal expansion coefficients. The C/C material coated with CVD-SiC withstood the 10 hour test program with no damage. For the SiC reinforced SiC composite, the test program was extended since this material promised good oxidation resistance. The maximum outer wall temperatures during the tests were 1050°C. The component was under operation for 90 hours and visual inspection showed no morphological defects.

Tests for the C/C- and the SiC/SiC-flame tubes are still continued. The project is planned to be completed with destructive tests of the tubes to obtain any structural changes in the materials.

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