MODEL COMBUSTOR TO ASSESS THE OXIDATION BEHAVIOR OF CERAMIC MATERIALS UNDER REAL ENGINE CONDITIONS

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ABSTRACT

A further increase of thermal efficiency and a reduction of the exhaust emissions of ground based gas turbines can be achieved by introducing new high temperature resistant materials. Therfore, ceramics are under international development. They offer excellent strengths at room and elevated temperatures. For gas turbine combustor applications, however, these materials have to maintain their advantageous properties under hostile environment. For the assessment and comparison of the oxidation behavior of different nonoxide ceramic materials a test rig was developed at the Institute for Thermal Turbomachinery (ITS), University of Karlsruhe, Germany.

The test rig was integrated into the high temperature/ high pressure laboratory. A ceramic model combustion chamber was designed which allowed the exposure of standard four-point flexure specimens to the hot combustion gas flow. Gas temperatures and pressures could be varied in a wide range. Additionally, the partial steam pressure could be adjusted to real combustor conditions.

The present paper gives a detailed description of the test rig and presents results of 100 hours endurance tests of ceramic materials at 1400°C. The initial strengths and the strengths after oxidation tests are compared. In addition to this, photographs illustrating the changes of the material's microstructure are presented.

NOMENCLATURE

ε	creep rate
K_{Ic}	critical stress intensity factor
m	mass flow
Ox	Oxidation
p	pressure
ρ	density
σ	strength
T	temperature
UHC	unburned hydro carbon

Subscripts:

H_2O	water/ steam
pri	primary
tot	total

INTRODUCTION

Future developments of ground based gas turbines will be aimed to an increase of the engine's thermal efficiency and a reduction of pollutant emissions. These goals can be achieved by higher turbine entry temperatures and a reduction of cooling air. This leads to high thermal loading of the components forming the hot-gas section of the engine: the combustor, transition ducts, turbine nozzles, and turbine blades. Because of this new high temperature resistant materials are required and consequently, ceramics are a matter of international development. They offer great potential for application in a high-temperature
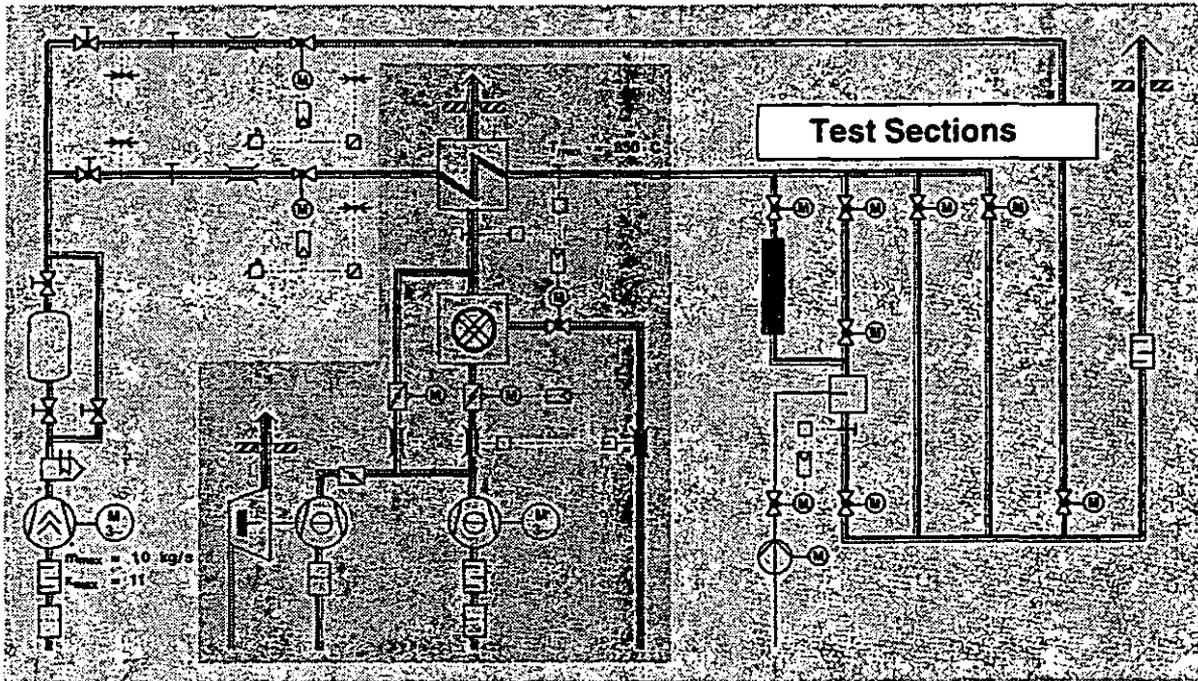
Fig. 1: Schematic of the high temperature/ high pressure laboratory at the ITS.

gas turbine. Their excellent high temperature properties qualify them to be employed in the design of static gas turbine components.

Especially, the application in combustor walls seems to be promising [Smith and Fahme, 1997]. Firstly, ceramics may allow an increase in the flame temperature with no reduction of the combustor's durability. Secondly, emission benefits can be achieved. The small amount of cooling air leads to reduced reaction quenching near the combustor walls which decreases the CO- and UHC-emissions in the exhaust. Additionally, in a well designed combustor the peak flame temperature can be reduced which results in lower NO_x production.

One of the main requirements for ceramic materials to be applied reliably in gas turbines is long-term stability with respect to mechanical properties at high temperatures. Recent investigations showed that there are ceramic materials available now which can survive more than 1000 h at high temperatures under laboratory conditions [Klemm et al., 1998; Wötting et al., 1998; Westerheide et al., 1997]. One feature of these materials, which is the main factor in stabilizing the mechanical properties, is their microstructural stability during long-term service at elevated temperatures.

Due to the severe environmental conditions in a gas turbine combustor, application of these laboratory tests is limited. The corrosive composition of the gas (H_2O) in combination with the high flow rate have great influence on the oxidation and corrosion behavior of ceramic materials, with the consequence of degradation of the mechanical properties and ultimately of the lifetime of the materials[Opila and Hann, 1997; Endo et al., 1994].

For that reason, a model combustor was designed at the Institut für Thermische Strömungsmaschinen (ITS) to assess the effect of oxidative corrosion on the long term mechanical behavior of ceramic materials under real engine conditions. In this combustor ceramic specimens could be exposed to the hot gas flow, since previous investigations showed that high speed gas flow appeared to be the reason for the disappearance of the protecting oxide layers, which resulted in a more rapid oxidation of the base material [Etori et al., 1997].

TEST FACILITY
At the ITS a high temperature/ high pressure laboratory is available. A schematic of the facility is shown in Figure 1. The test facility is capable of supplying different test sections with preheated, non-vitiated air. The heat exchanger installed allows air temperatures up to 850 °C, the maximum mass flow rate is 1 kg/s, and the compressor pressure ratio is 10.

The model combustor test section, which has a total length of 3000 mm, consists of five parts. As illustrated in Figure 2, in the first part the air mass flow is divided into the inner, primary air mass flow and the secondary air mass flow. This is necessary to avoid critical high wall temperatures in the combustor region. The relatively cold secondary air flow protects the outer metal containment. In the second part, the primary air mass flow is determined by an installed orificemeter. To reach a homogenious flow field in the primary pipe an appropriate forerun to the aper-
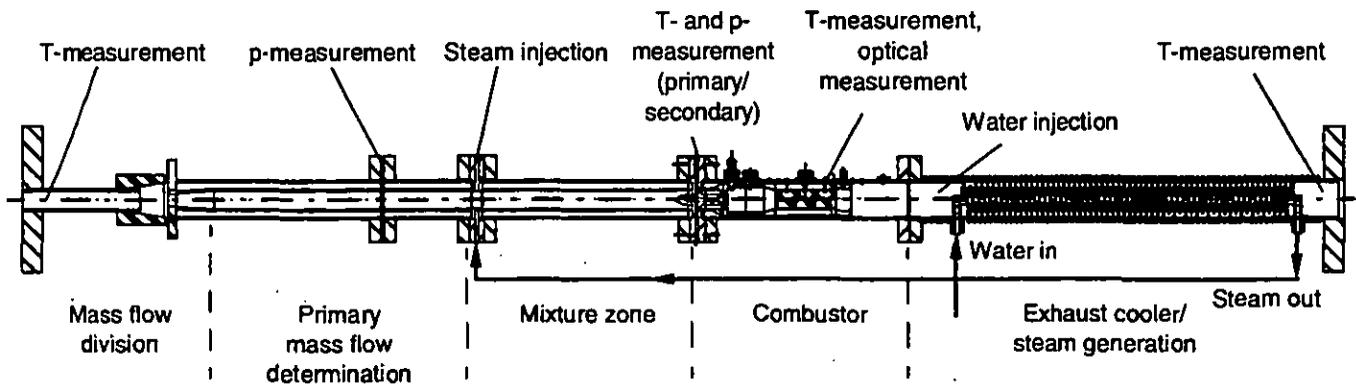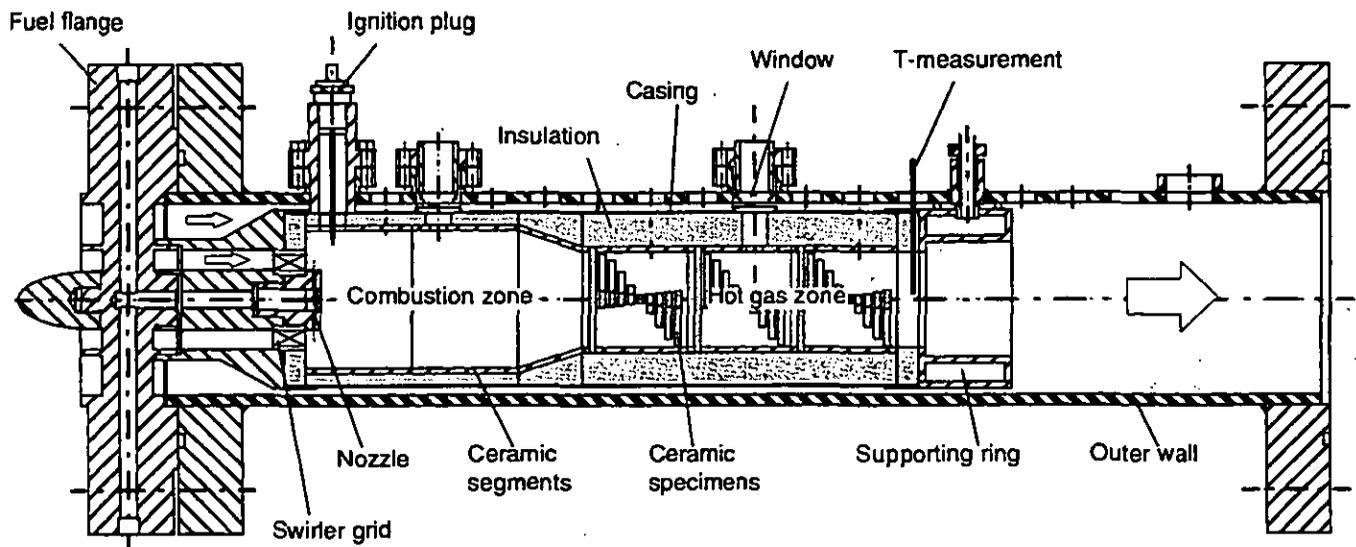
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Fig. 2: Schematic of the test section.

Fig. 3: Cross section of the model combustor.

ture had to be planned. The mixture zone is necessary for uniform mixture of steam and air mass flow. Steam has to be injected to reach realistic steam partial pressures in the model combustor, since this has strong influence on the oxidation behavior of ceramic materials [Opila and Hann, 1997]. The steam is generated in a heat exchanger consisting of helically coiled tubes. It utilizes the heat of the exhaust gases and is included into the last part of the test section, which operates as a combustion gas cooler (which is appreciated since the exhaust gas temperature has to be reduced anyway before entering the exhaust ducts). With a flowmeter, the amount of feed water for the steam generation can be determined. Fully desalinated water is used to avoid harmful chemical elements being added to the hot gases. To reach adequate exhaust temperatures, additional water injection is possible.

In the following section, the model combustor integrated into this facility is described in detail.

MODEL COMBUSTOR

The construction was greatly based on the comprehensive experiences gained from previous and present research activities at the ITS in designing ceramic gas turbine components [Filsinger et al., 1997], combustion chambers [Maier et al., 1997; Meisl et. al., 1997], and ceramic combustors in particular [Pfeiffer et al. 1991]. As shown in Figure 3, it was built as a hybrid construction, offering the advantages discussed in Münz et al. [1996]. The ceramic rings forming the combustor and hot gas zone were manufactured at the Fraunhofer-Institute of Ceramic Technologies and Sintered Materials in Dresden (IKTS) and consisted of sintered silicon carbide (SSiC). The rings were segmented in axial direction to reduce the temperature gradients in the single parts. The combustion zone was 130 mm in length and the diameter was 80 mm. Thereafter, the hot gas zone with a total length of 150 mm was connected. In that zone it was possible to expose standard four-point-flexure specimens (50 mmx3.8 mmx3.0 mm) to the hot gas stream.
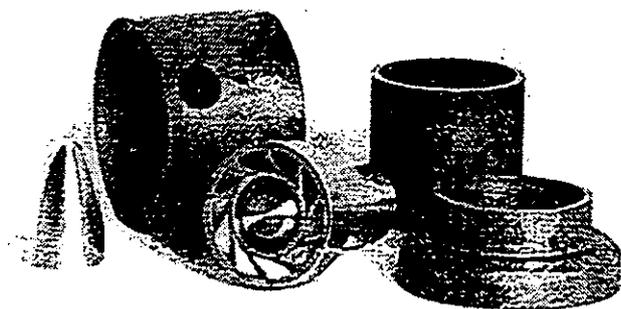
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Fig. 4: Ceramic combustor elements (from left to right):
combustor nose, ring element combustion zone, swirling
grid, ring element hot gas zone, transition element.

Ignition was initiated by a conventional spark plug. Natural gas served as fuel which is compressed to the pressure required. It was supplied through the fuel flange and an in-house designed nozzle. The primary air was lead through a swirling grid to stabilize the flame. Figure 4 shows some elements which form the ceramic combustor.

As mentioned before, the preheated total air mass flow is divided. The secondary air is lead through a narrow ring channel between the combustor's casing and the containment. The relative amount of primary and secondary air is about 1:4 and, therefore, the high heat transfer in the ring channel guarantees moderate outer wall temperatures. The insulation between the combustor's casing and the ceramic rings helps to reduce the heat loss in the flame tube, and adjusts the mismatch of thermal expansion between the metal and SSiC segments. Silicate fiber fabric and alumina silicate fiber-vacuum-formed products serve as insulating materials. These materials have proved their ex-
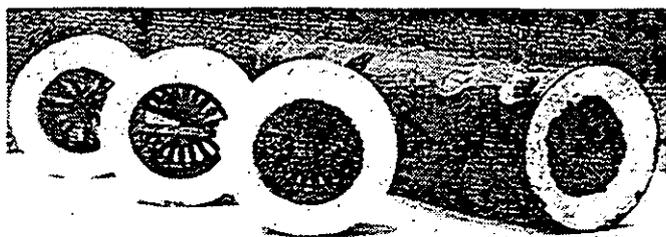

Fig. 5: Left: insulated ceramic segments which form the
hot gas zone and support the ceramic specimens;
right: ceramic combustor in metallic casing.

cellent properties under severe conditions in a different combustor test rig installed at the ITS [Filsinger et al. 1997]. The casing is supported downstream by a water cooled element mounted to the outer wall.

Probes were installed for the determination of air temperatures and pressures in the flow channels. At the combustor's entry the primary and the secondary flow was qualified by static

and total pressure, and the total air temperature which is measured by using NiCr/Ni-thermocouples. At the exit of the hot gas zone, the gas temperature was measured with PtRh/Pt-thermocouples. To analyze the composition of the exhaust gas, it was possible to insert a multi-hole emission probe at the end of the hot gas zone. The analyzing system is capable of determining concentrations of NO, NO_2, N_2O, C_xH_y, CO_2, CO, SO_2 and O_2 continuously. To facilitate temperature measurement using optical pyrometers, the combustor was equipped with sapphire-windows that are translucent for heat radiation. On three positions the temperature on the surface of the installed ceramic specimens can be specified by a two-wavelength pyrometer, shown in Figure 6. By doing this, the thermal loading of the test bars could be adjusted and regulated. Data aquisition of the test facility and the model combustor was fully computer-controlled.

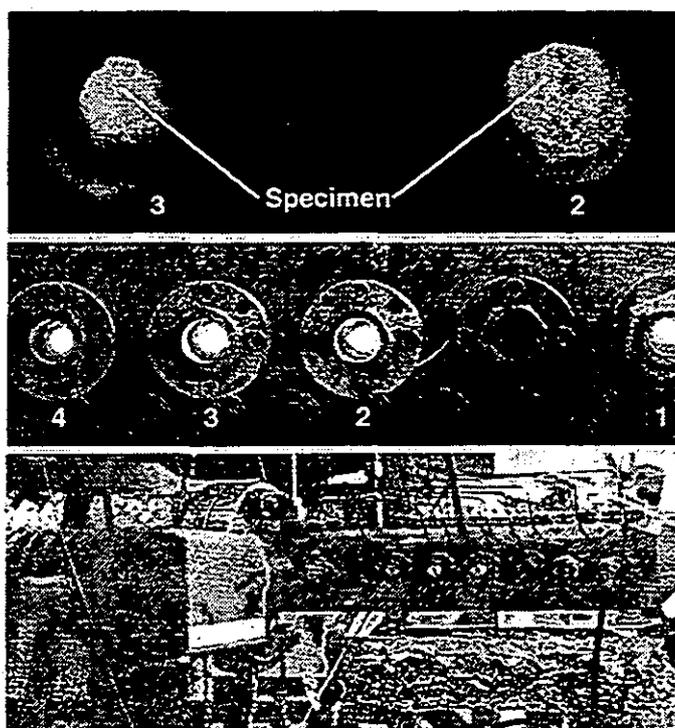In the hot gas section, 30 ceramic test bars could be exposed

Fig. 6: Test section in operation (flow from right to left).
Top: enlighted windows; combustion zone 1, hot gas zone
2-4. Bottom: combustor and pyrometer (foreground).

to an engine like environment. They were supported by ceramic rings, which contained rectangular openings to hold the specimens, which were stuck radially through the circular flow channel (see Figure 5). Typical candidate materials (silicon-based) were chosen for the oxidation tests.

TEST CONDITIONS

As mentioned before, it was possible to vary pressure and temperatures in the test rig. Additionally, the partial steam pres-
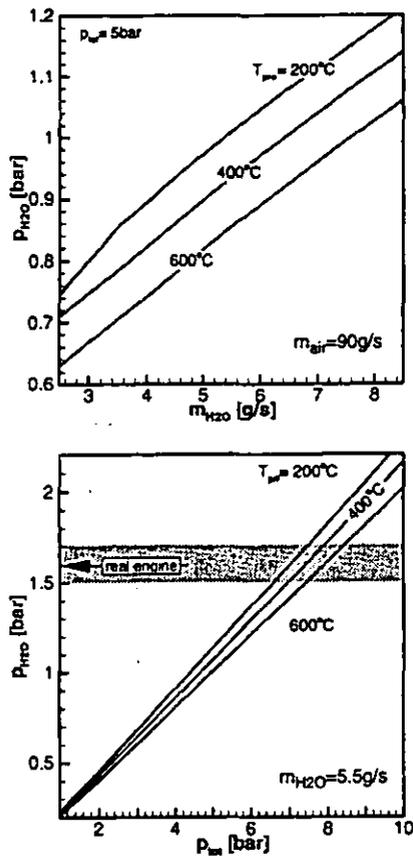
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Fig. 7: Possible variation of partial steam pressure for different primary air temperatures. Top: Influence of added mass of steam; Bottom: Influence of total pressure.

sure could be varied by steam injection. In Figure 7 the possible calculated variance is illustrated. The equilibrium calculations were performed with the Chemkin II software package [Kee et al., 1989], under the assumptions of a complete and adiabatic chemical reaction mechanism [Miller and Bowman, 1989]. Prescribed values for these estimations were the flame temperature of 1400°C and a flow speed of 50 m/s in the hot gas zone. The upper part of Figure 7 shows the partial steam pressure (p_{H2O}) versus the amount of added steam mass flow (m_{H2O}) for different primary air temperatures T_{prim}. The influence of the total pressure in the test section (p_{tol}) can be seen in the lower portion of Figure 7.

As expected, increasing the primary air temperature led to lower partial steam pressures, since the amount of natural gas had to be reduced if the same flame temperature was maintained. Figure 7 also illustrates that the total air pressure and the amount of added steam were almost correlated linearly to the partial steam pressure.

For a comparison of the gas atmosphere with the one prevailing in a real engine combustor, the partial steam pressure was calculated. As realistic boundary conditions, a pressure ratio of 16.8, a combustor inlet temperature of 400°C, a flame tempera-

ture of approximately 1450°C, and an air-to-gas ratio of 2.0 were assumed. Under these conditions the partial steam pressure attains values of about 1.6 bar (see shaded area in Figure 7).

For the presented results of the hot gas tests a thermodynamic situation was determined. Table 1 gives a short summary of the conditions established in the test section for the 100 hours endurance tests. This indicates that in the hot-gas zone of the test rig nearly realistic conditions, typical for a gas turbine combustor, could be adjusted. Only the partial steam pressure of water vapor had to be limited due to flame stability difficulties.

Pressure	5 bar
Air mass flow	90 g/s
Flow speed	50 m/s
Partial steam pressure	900 mbar
Temperature on specimen 7	1400 °C

Tab. 1: Test conditions.

During the tests, the thermal power (amount of natural gas) was regulated by the adjustment of the surface temperature on the specimen to the required value. This control parameter was measured by the two-wavelength pyrometer. Surface temperatures were measured on the 7th, 17th, and 27th specimen counted along the stream direction. In the hot-gas zone, a temperature loss of 50 K was observed in axial direction, which was attributed to heat losses in the channel. The three measuring positions allowed the determination of specific temperature loading of each specimen in the test section.

EXPERIMENTAL RESULTS

The results of the rig tests will be described by comparing three ceramic materials: a Si_3N_4 material, a $Si_3N_4/MoSi_2$ composite with Y_2O_3 as a sintering aid and, a S SiC material (solid-state-sintered SiC with B and C as sintering additives). Relevant properties of the materials are summarized in Table 2. All materials exhibit excellent high-temperature stability.

From the burner rig tests, two general problems were identified: the microstructural stability of the bulk material and the stability of the oxidation surface.

With respect to the microstructural stability of the bulk material, the results of the rig tests confirmed those of oxidation tests performed and reported previously [Klemm et al., 1998; Klemm et al., 1997, Klemm et al., 1997]. During the rig tests, the microstructure changed due to oxidation processes that were dependent on the diffusion of oxygen into the upper region of the bulk, and reaction of the oxygen with the grain boundary phase. The S SiC material was found to have a low rate of oxygen diffusion. All of the oxygen reacted at the interface between the oxidation layer and the bulk, which consequently was not si-

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gnificantly changed or damaged. As in oxidation tests performed in previous studies, the microstructures of the two Si_3N_4 materials were found to be different after the tests. During the tests at elevated temperatures some of the diffusing oxygen penetrated into the bulk material. The reaction of this oxygen caused typical oxidation damage to occur to such an extent that pores were formed in the middle of the material (as can be seen in the Si_3N_4 material in Fig. 8a).

Material	ρ g/m^3	σ_{RT} MPa	σ_{1400} MPa	K_{Ic} MPa/ m$^{1/2}$	ε h^{-1}	Ox. mg/cm^{-2}
SSiC	3.18	430	430	2.8	1.5×10^{-6}	1.2
SN	3.29	890	730	8.2	1.5×10^{-5}	2.0
SN/Mo	3.47	830	700	8.2	1.8×10^{-5}	3.0

Tab. 2: Mechanical properties of the materials investigated: σ_{RT} and σ_{1400} (four-point flexure strength at room temperature and 1400°C), K_{Ic} (SENB t=0.15 mm, ε: 200 MPa, 1400°C, calculated using the deflection in the inner span of four-point bending cell between 90 and 100 h, the nonlinearity of the elastic stress distribution in the beam and the difference of the materials under tension and compression were neglected); Ox: weight gain after 2500 h oxidation at 1500°C in air.

The $Si_3N_4/MoSi_2$ composite material showed a different behavior. Due to a changed oxidation mechanism (formation of additional Si_2ON_2 instead of SiO_2), the microstructure of this material was not different after the test (Fig. 8b). The different behaviors of these materials were reflected in the residual flexure strength of the specimens after the rig test as shown in Table 3.

Due to microstructural degradation in the bulk, the residual strength of the Si_3N_4 material was found to be only 45% of the strength in the hot-pressed condition. This type of microstructural degradation was not observed in the SiC material or the $Si_3N_4/MoSi_2$ composite; consequently, these materials had a high residual strength after the test.

Material	σ_{test} MPa	σ_{test}/σ_0 -	Δm %
SSiC	350	0.81	10
SN	400	0.45	8
SN/Mo	660	0.80	7

Tab. 3: Residual strength and weight loss after the rig exposure (100 h, 1400°C); each value represents the average for at least 4 specimens.
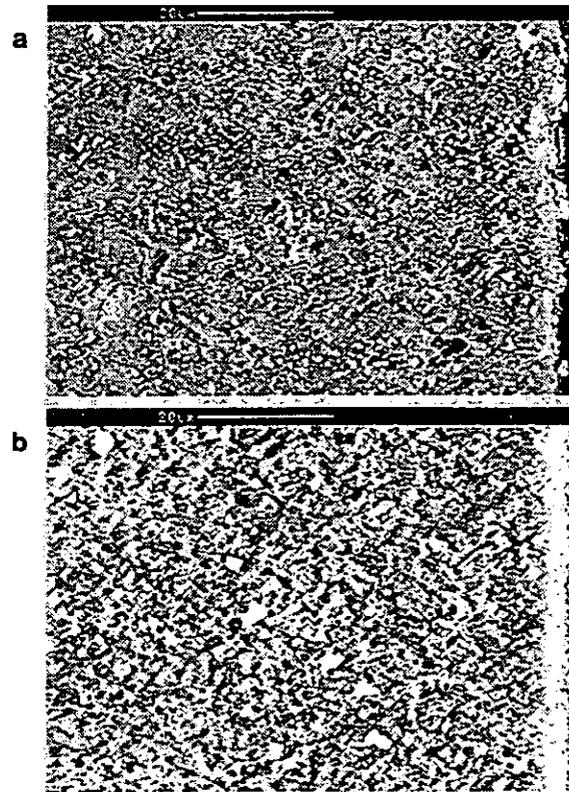

Fig. 8: SEM images of polished cross sections of the Si_3N_4 material (a) and the $Si_3N_4/MoSi_2$ composite (b) after the rig test at 1400°C.

An important requirement for the application of nonoxide ceramic materials in gas turbine components is the formation of a stable, protective oxidation layer. Silicon-based materials, such as SiC, Si_3N_4 and $MoSi_2$, form an oxidation layer consisting of SiO_2, which is very effective due to the low diffusivity of oxygen in SiO_2. This protective layer is effective up to temperatures of about 1600°C in air. In a gas turbine environment, however, the stability of SiO_2 at elevated temperatures is limited [Opil a and Jacobson, 1997; Opil a and Jacobson, 1998]. All materials investigated exhibited a significant weight loss after the rig test (Table 2). The reason for the weight loss is enhanced formation and evaporation of SiO or $Si(OH)_x$ due to the presence of water and the high gas flow. The protective oxidation layer, which remained undamaged during oxidation in air, was damaged. This, consequently, led to accelerated oxidation of the materials during the rig test in an environment similar to that in gas turbines. The result of the evaporation processes is demonstrated by comparison of the surface of the $Si_3N_4/MoSi_2$ composite after oxidation in air (Fig. 9a) to that after the rig test (Fig. 9b).

Whereas the oxidized specimen had a typical smooth oxidation surface, consisting of $Y_2Si_2O_7$ crystallites in a mainly glassy layer of SiO_2, the material subjected to gas turbine con-
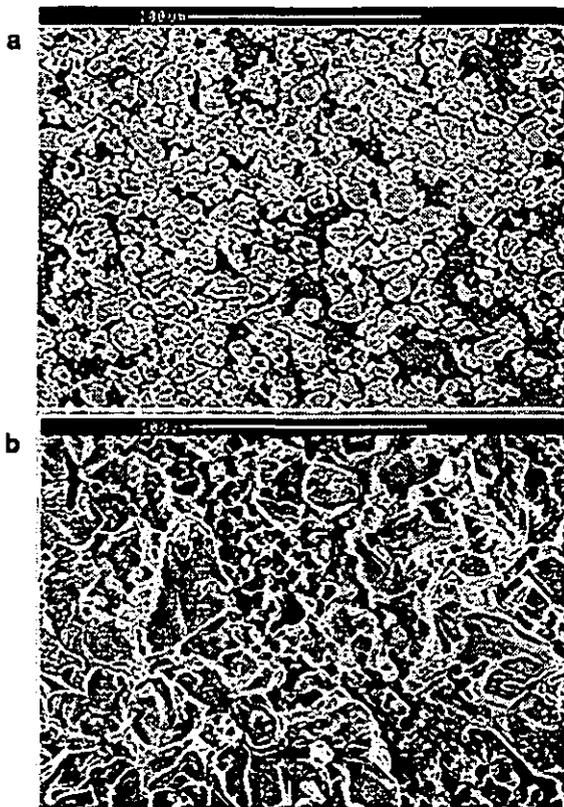
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Fig. 9: Comparison of the surface layer of a $Si_3N_4/MoSi_2$
composite after oxidation in air (a)
and after a test in gas turbine conditions (b).

ditions had a considerably different surface. Due to the evaporation processes, the protective SiO_2 layer was destroyed during the test. SiO_2 was leached out, leaving a very rough layer consisting only of $Y_2Si_2O_7$ crystals. The $Y_2Si_2O_7$ was found to be more stable in this environment, and may have had a stabilizing effect on the evaporation of SiO_2. A similar surface was found in the Si_3N_4 material. The S SiC was found to experience the largest weight loss. This was a consequence of the formation of a completely unprotective oxidation layer consisting only of SiO_2 and no silicates.

SUMMARY

The present paper gives a detailed description of the model combuster integrated into the high temperature/ high pressure laboratory at the Institute for Thermal Turbomachinery, University of Karlsruhe (TH), Germany. Its simple construction allows the use of ceramic components in the hot gas path to realize high gas temperatures. In the test rig, a wide variation of test conditions is possible. Temperature, pressure, air mass flow, and partial steam pressure can be varied independently. This enables the assessment of the oxidation behavior of ceramic materials under real engine conditions.

Results for 100 hours endurance tests at 1400°C were shown. The corrosion behavior of three ceramic materials: a Si_3N_4 ma-

terial, a $Si_3N_4/MoSi_2$ composite with Y_2O_3 as a sintering aid, and a SSiC material were compared. A good microstructural stability after long term oxidation in air and the rig test was observed in the SiC material and the $Si_3N_4/MoSi_2$ composite. All materials suffer degradation of the protective SiO_2 oxidation layer caused by evaporation processes. These evaporation processes could be reduced by the insitu-formation of more stable silicates ($Y_2SiO_2O_7$) in the additive containing Si_3N_4 materials during oxidation.

ACKNOWLEDGMENTS

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