Abschlußbericht
zum F&E Projekt COST 510

Hochleistungswerkstoffe für Temperaturen oberhalb 1500°C – Entwicklung von Testmethoden

Teilprojekt: Entwicklung der Laserheiztechnik

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DEVELOPMENT OF LASER HEATED OVEN AND SURFACE TREATMENT OF REFERENCE SAMPLES WITH LASER

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ABSTRACT

A laser heated furnace has been developed, where materials can be heated up by laser beams to high temperatures in vacuum or different gas atmospheres. With a special equipment it is possible to heat up the samples either from one side or from both sides simultaneously. The emissivity can be observed either orthogonal to the sample surface or in dependence of different angles.

For the development of reference samples first investigations have been carried out. The experiments showed that remelting of plasma sprayed ceramic coatings on high temperature materials is possible if a suitable combination of coating and base material is used. To judge on the quality a detailed investigation of the processed samples is necessary.

1 INTRODUCTION

The knowledge of thermophysical properties is essential to calculate temperatures of thermally loaded components. These are important to estimate the lifetime of a component which is limited by the extreme temperature or to judge mechanical stresses caused by temperature gradients or rapid temperature changes.

Some of the most important properties are the thermal conductivity, thermal diffusivity and total emmitance. In special the knowledge of radiative as well as conductive properties are of great importance for the layout of thermally loaded components in many fields of industry. All these properties are essentially dependent on the structural composition of the material.

The principal of thermal diffusivity measurements consists of a variation of the temperature of the sample by a modulated heating source [1]. Thus the thermal diffusivity can be calculated. For measurements of thermal emissivity an isothermal heating is necessary and for measurements of heat flux a well known temperature gradient between both sides of a disc shaped sample should be installed [2].

For all these measurements at high temperature materials an heating device is needed, which can heat a sample to a very high surface temperature, up to the temperature shortly below the melting point of the materials at least up to the temperature above 1500°C. The heating should be possible in different atmospheres and vacuum.

2 DEVELOPMENT OF LASER HEATED OVEN

2.1 New heating technique

For the emissivity measurement of materials at high temperatures a couple of different heating techniques have been developed and applied by different researchers in different countries up to now. These different techniques are resistance heating, electron beam heating [3], surface induction heating, heating with quartz halogen lamps [4], solar heating [5], cavity induction heating and microwave heating [8]. Also laser heating in combination with resistance heating of samples was applied [6]. In a couple of cases the heating method in the high temperature range can only be applied in vacuum or certain atmospheres. Very often the observation of the specimen can not be done from the same side as the heating takes place. In many cases the heating has to be stopped during the observation of the sample for the radiation measurements, because the radiation from the heating source is higher than from the sample surface and in the same wavelength region where the measurements take place. Nearly always, when simultaneous measurements have to be done, the heating is not isothermal.

Fig. 1 shows the schematic drawing of the new designed laser heated furnace. The disc shaped specimen is heated up in a temperature isolated chamber by either one or two laser beams coming from two different sides onto the specimen surface.

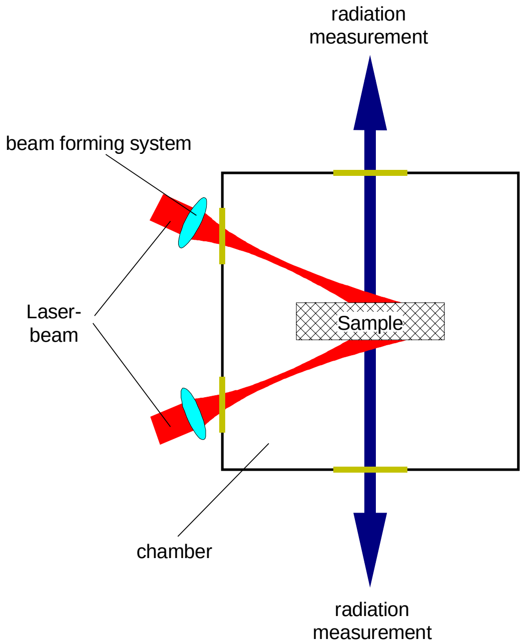


***Fig. 1:** Schematic drawing of the laser heated oven*

The isothermal heating by using two laser beams from two sides is especially necessary if semitransparent samples have to undergo radiation tests. At pyrometric radiation testing methods a gradient in the observed region leads to wrong results, because the measurement point can not be determined exactly. So if both sides of the semitransparent specimen have the same temperature the measured radiation will be independent of the point in z-direction where the temperature is measured.

2.2 Technical design of the furnace

The technical design of the furnace is shown in two different views in the following two figures (see Fig. 2 and Fig. 3).

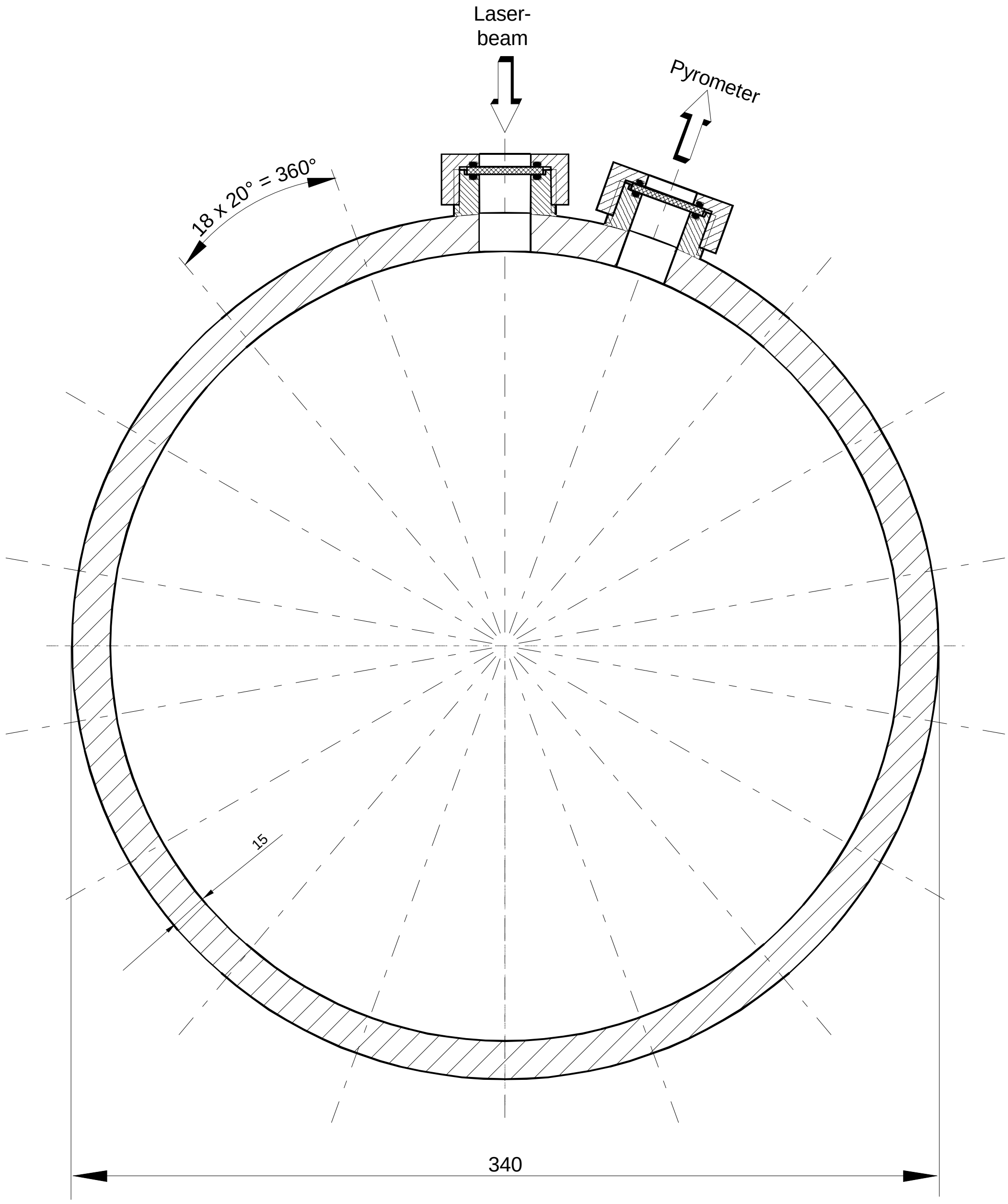


Fig. 2: *Technical design of laser heated furnace*

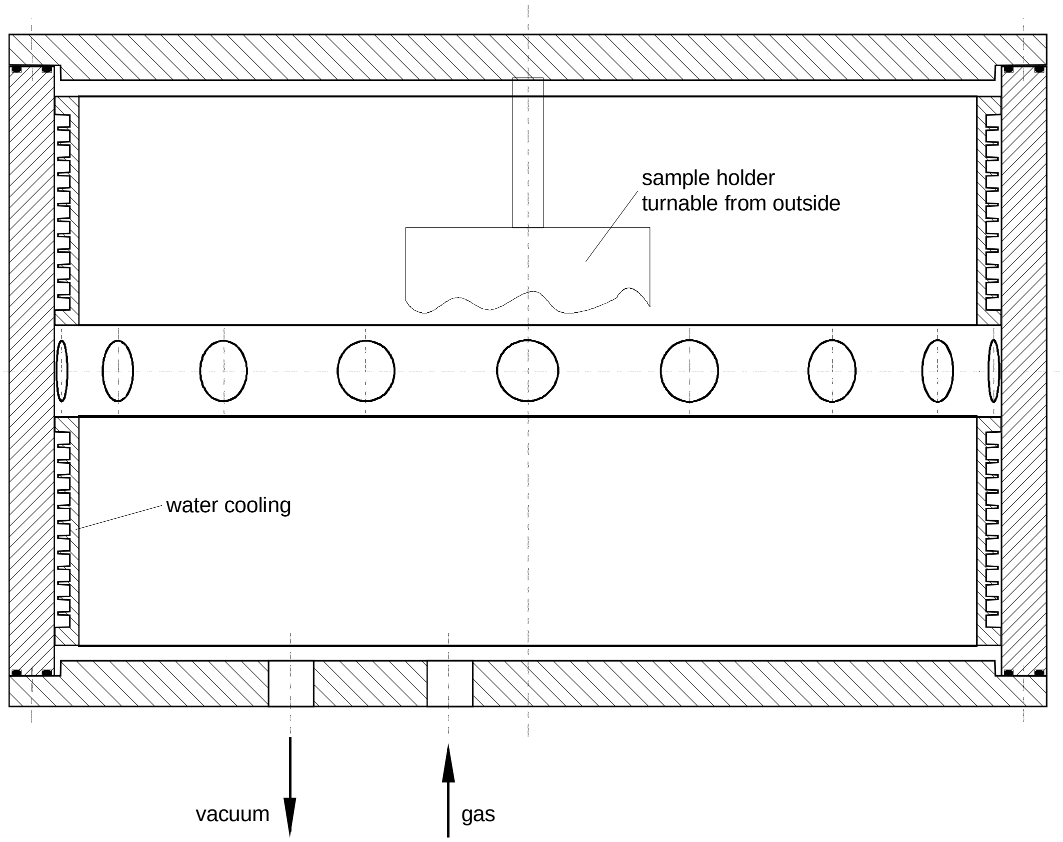


Fig. 3: *Technical design of laser heated furnace*

Around the furnace 18 windows are installed, where each can be used either for the laser heating device or for the equipment for emissivity measurements. The chamber can be evacuated or filled with different atmospheres e.g. inert gases (N_2, Ar, etc.) or reactive gases (O_2, etc). The integrated water cooling system will guarantee a constant temperature of the outside surface of the oven.

2.3 Design of the beam guiding and beam forming system

For heating up the samples two different lasers (CO_2-laser with 10600 nm and Nd:YAG-laser with 1064 nm) with two different wavelengths are available. Because of the two different wavelengths, different materials can be heated which may be transparent at one of the two wavelengths. CO_2-lasers are useful for heating ceramic materials, whereas Nd:YAG-laser would be better for metallic materials due to the better absorption.

For the CO_2-laser the beam guiding system has to be done with mirror optics and the beam forming can be done either with focusing mirror optics or with special glass lenses. The work can be done with a 750 W Rf-excited CO_2-laser. The output power can be modulated by an analogue signal together with a special designed device by direct-controlling of the high frequency generator up to 6,5 kHz.

In normal the Nd:YAG-laser is used, which is guided to the insulation chamber with optical fibers. The beam forming is made with normal glass lenses. Different beam diameters (4.6, 5.6, 7.7, 9.5 and 11.5 mm) with a flat top intensity profile (see Fig. 4) on the sample surface are available. By using a beam splitter it is possible to split the laser beam in two beams, each in its own fiber, with a fixed ratio of 50% of the output power. The maximal output power is 2 kW cw (continuos wave) and can be modulated with a maximum frequency of 500 Hz by an analogue signal.

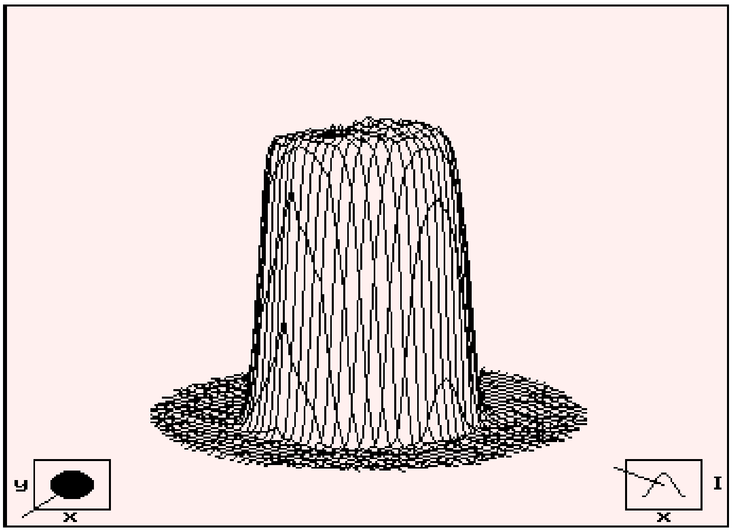


Fig. 4: *Nd:YAG intensity beam profile at surface of specimen*

For the isothermal heating of the samples the surface temperature has to be controlled online by a pyrometer. This allows to measure and protocol the surface temperature to control the laser power, so that the process can be kept stable (see Fig. 5).

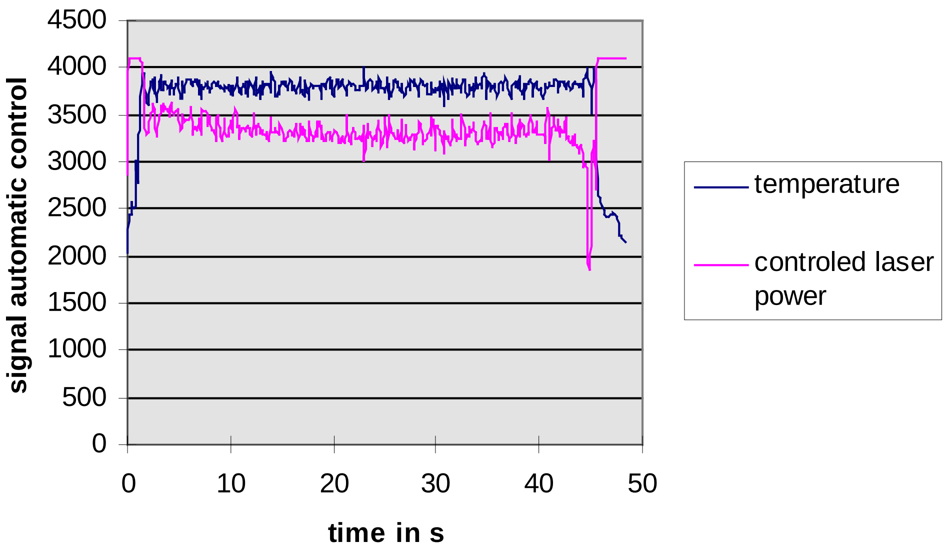


Fig. 5: *Automatic surface temperature control with pyrometer (set value=3800)*

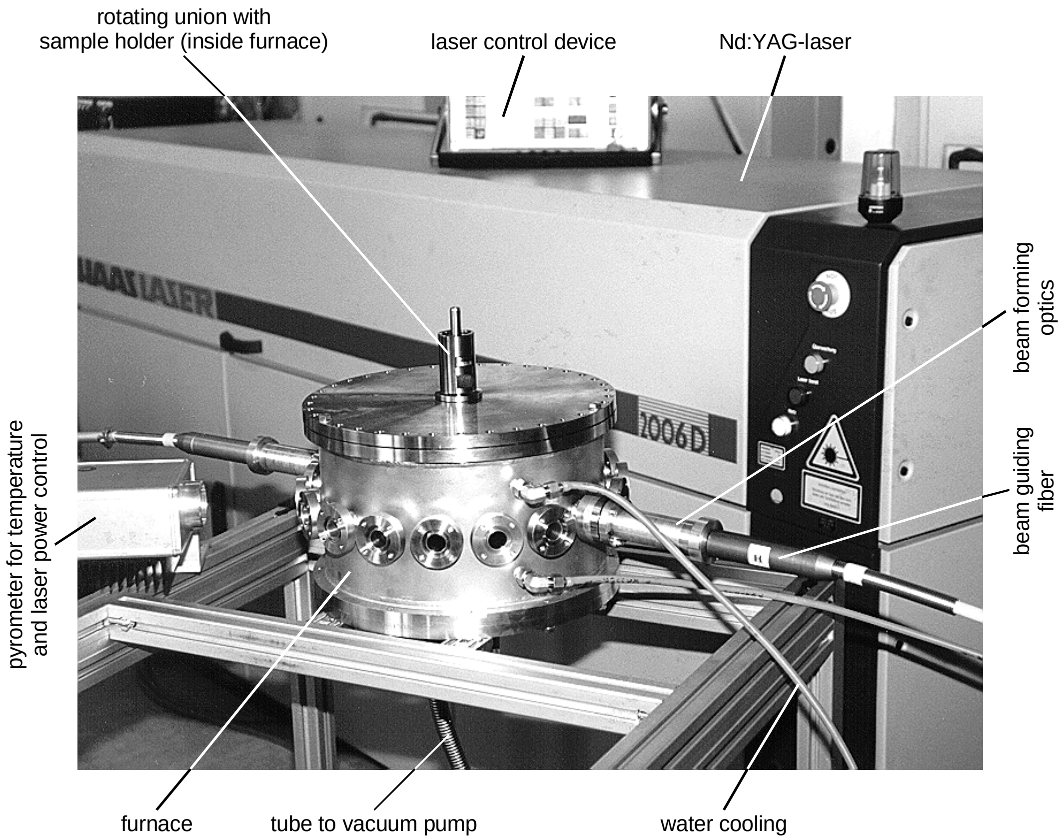


Fig. 6: *Experimental setup of the laser heated furnace*

Fig. 6 shows the experimental setup for the laser heated furnace. It consists of the furnace itself, the rotating union with the mounted sample holder (inside furnace), the Nd:YAG-Laser with its control device, two beam guiding fibers (core diameter of 600 µm) and lenses, the pyrometer for temperature and laser power control, the water cooling system and the device for chamber evacuation.

For the measurement of radiation a new measurement device which is developed by KE (Institut für Kerntechnik und Energiewandlungssysteme e.V., Stuttgart) will be applied at the oven. This device is a combination of a multiwavelength pyrometer and a laser source with two different wavelengths. With such an instrument the emissivity ratio at the two laser wavelengths can be determined and used to convert the measured radiance temperatures into the true surface temperature [7]. The schematic view of the experimental setup is shown in Fig. 7.

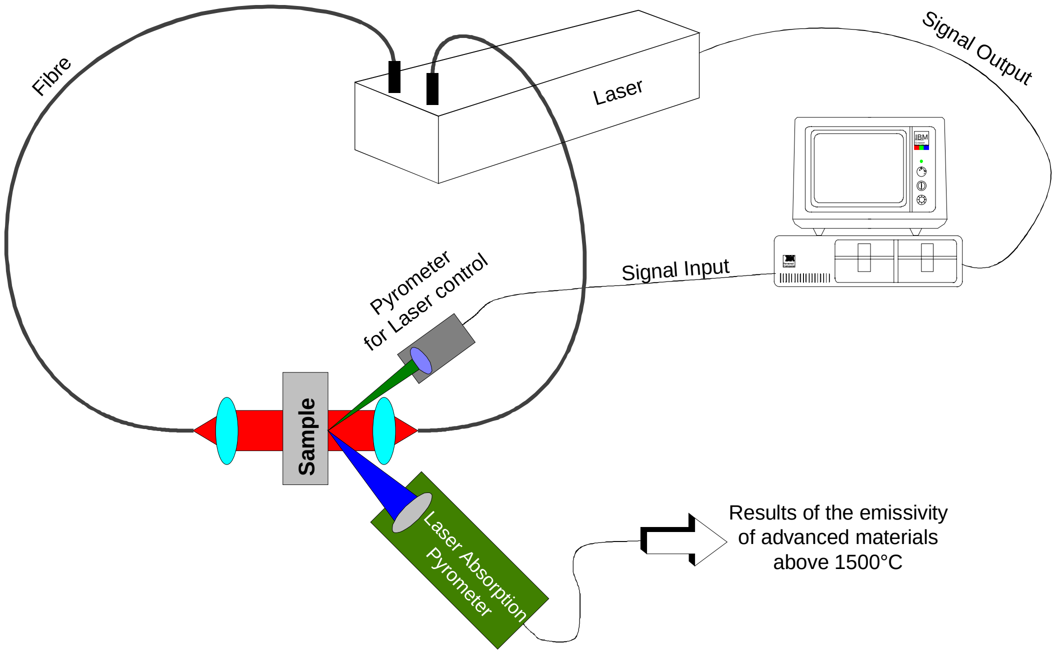


Fig. 7: *Schematic view of the experimental setup for laser heating and radiation measurement*

3 FINITE ELEMENT CALCULATION OF ISOTHERMAL SPECIMEN HEATING

For observing the emissivity of reference samples it will be necessary to have a part in the sample with isothermal conditions where the measurements will take place. Therefore calculations have been made with the method of finite elements by using the software PERMAS (calculation) and IDEAS (modeling).

The sample was defined as a disc shaped specimen made of steel with a diameter of 30 mm, a thickness of 5 mm and a temperature of 20°C at the begin of the heating.

The heating source was modeled as a temperature profile with 1500°C and a diameter of 5 mm. The temperature source was set directly into contact with the specimens surface. Convection has not been taken into account.

First calculations were carried out with a one-sided heating source. Fig. 8 shows the temperature gradient of the sample after heating times of 0.5, 5, 10 and 20 seconds. After 20 seconds heating time the sample still has a temperature difference at both sides of at least 250 K which may not be acceptable for measurements. The temperature in radial direction is much bigger.

In further calculations both sides of the specimen were exposed to the temperature simultaneously. In Fig. 9 and in Fig. 10 the temperature gradient in the specimen is shown after a heating time of 5 seconds and after 3 minutes. After 3 minutes heating time the temperature difference from the surface of the sample to the middle inside the sample is only 1,3 K. The difference on the surface from the center to the border is about 9 K.

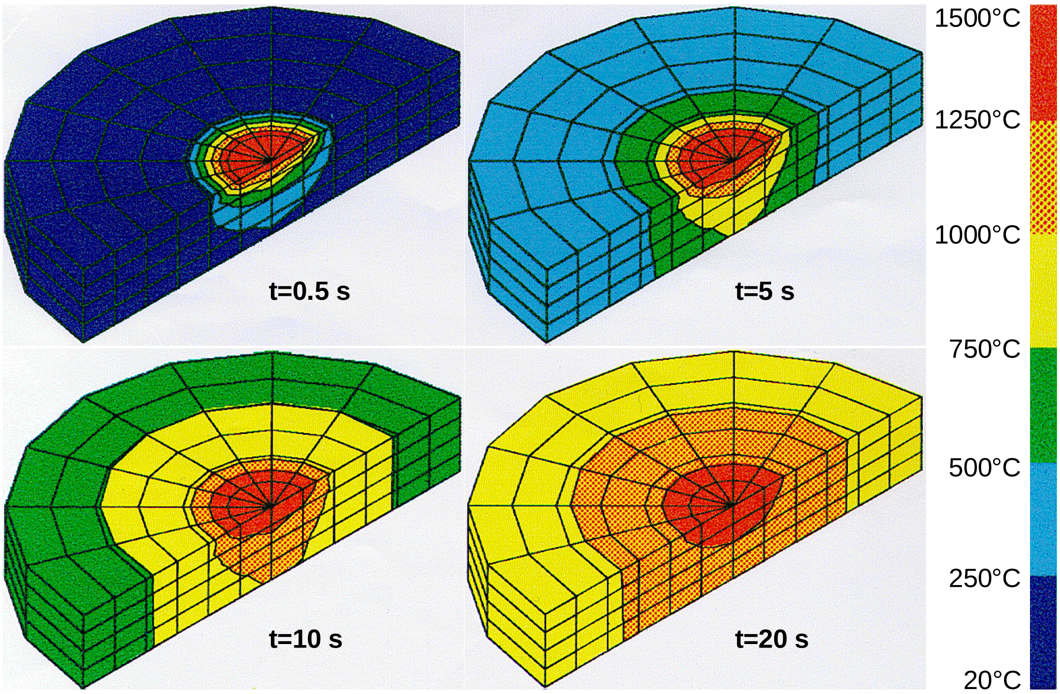


Fig. 8: *Temperature gradient in a one-side heated sample*

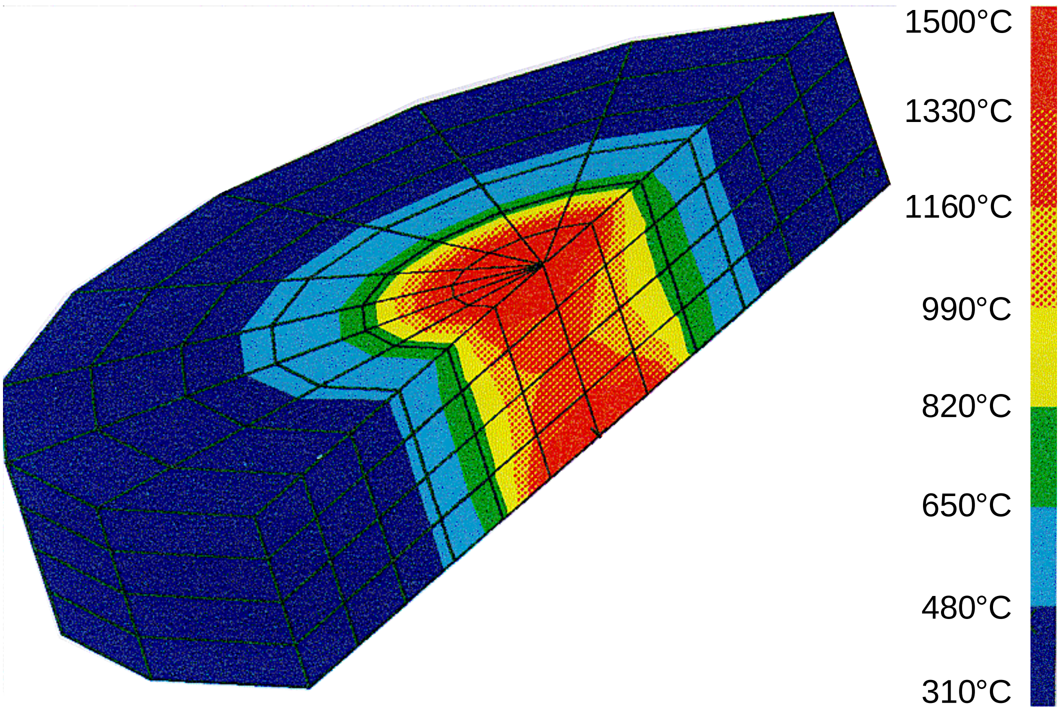


Fig. 9: *Temperature gradient in a two-side heated sample; heating time=5s*

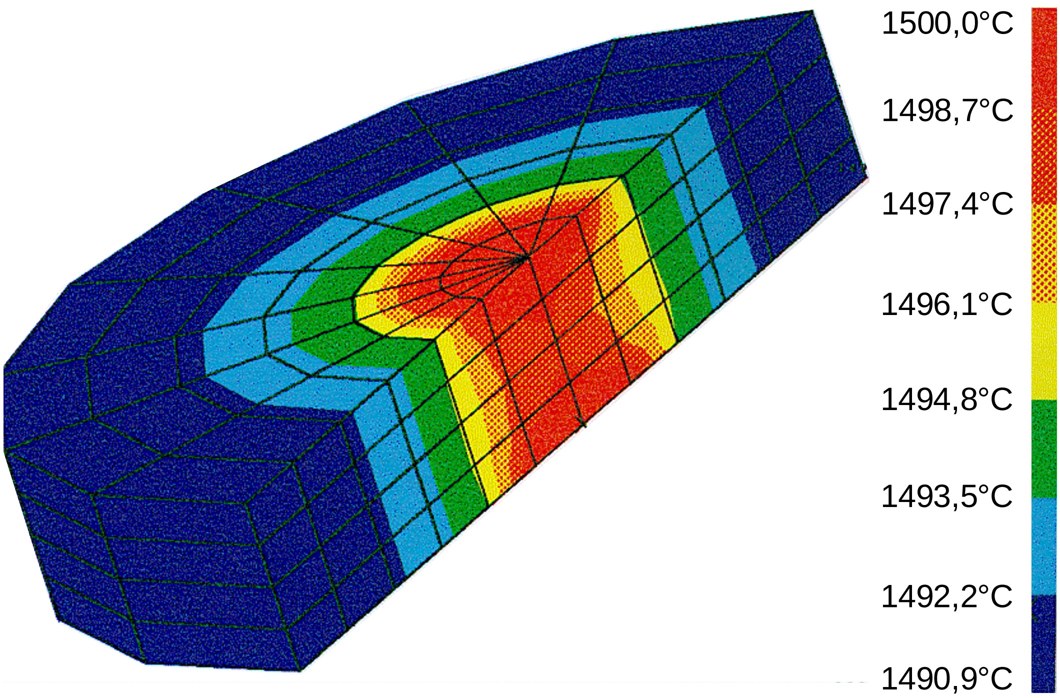


Fig. 10: *Temperature gradient in a two-side heated sample; heating time=180s*

When using a two-sided heating technique the small temperature differences from surface to the middle and from center to the border are acceptable for emissivity measurements.

4 SURFACE TREATMENT OF REFERENCE SAMPLES WITH LASER

4.1 Development of new reference samples

A very important aim is the development of reference samples for the measurement of emissivity at temperatures above 1500°C.
Plasma sprayed coating materials like Al_2O_3, CeO_2, ZrO_2 show no sufficient protection on high temperature (HT) materials. These materials tend to oxidation because of their high diffusibility for oxygen and their low density. The aim of the tests, carried out at Institut für Strahlwerkzeuge (IFSW), University of Stuttgart, is to improve the density of the coatings by surface treatment of reference samples with laser. Laser remelting of ceramic coatings causes microstructural changes and therefore increases the resistance against hot-gas corrosion and thermal shock.

4.2 Experimental set up for the remelting

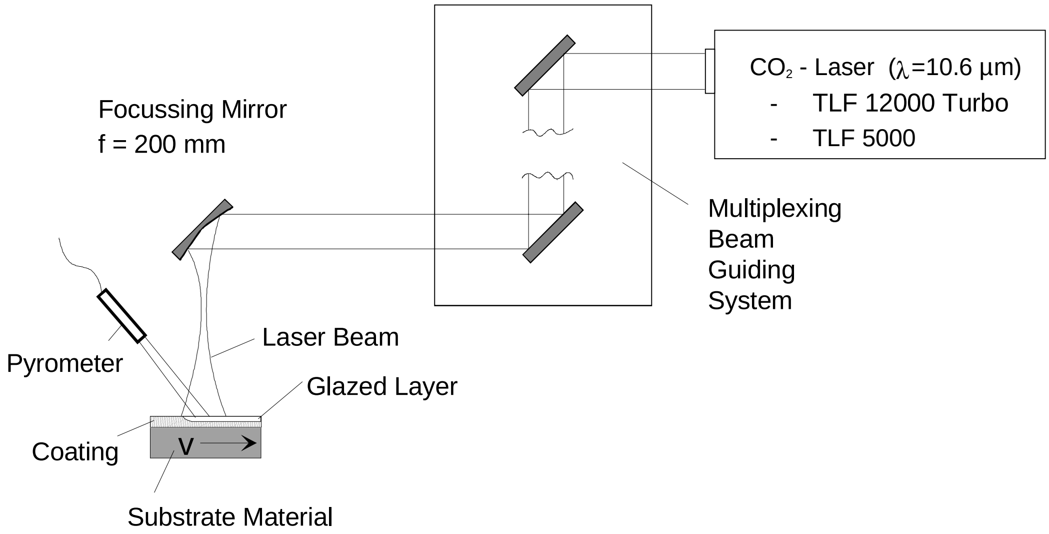


Fig. 11: *Experimental setup for laser remelting*

Tests were carried out to remelt plasma sprayed ceramic coatings by using CO_2-lasers (see Fig. 11). The lasers used were a 5 kW cw (continuos wave) system and a 12 kW cw system. The output power was controllable in a range of 5 to 100% of the maximum power. The laserbeam was focused on the sample by using a mirror optic with a focal-length of 200 mm. The beam diameter on the sample was 5,3 mm and the beam intensity profile is shown in Fig. 12. The temperature of the melt-pool was observed by using a pyrometer.

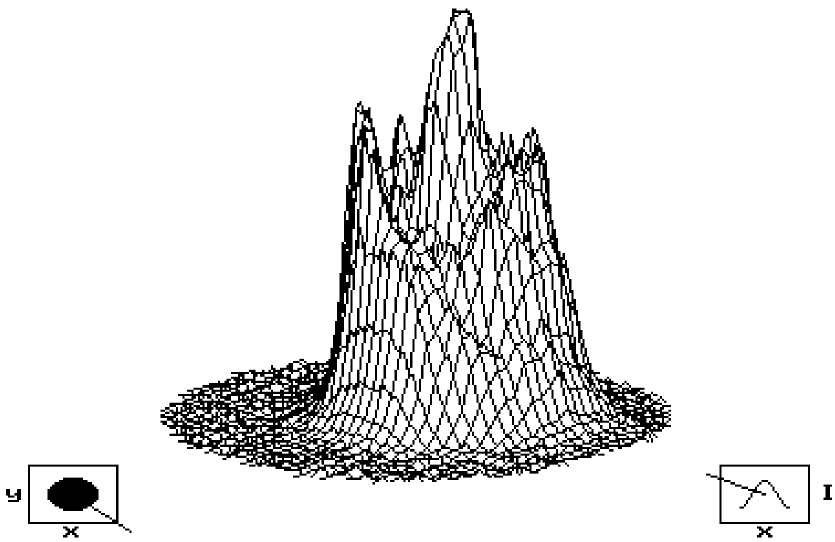


Fig. 12: *Intensity profile of CO_2-laser beam in focus*

4.3 Substrate and coating materials

DASA placed at disposal various combinations of substrate and coating materials. The coating was done by plasma spraying. Three main groups of samples can be distinguished.

- Single layer coating on C/SiC „*Carbosil*“:
  $Zr0_2$ „*Zirconium oxide*“
  Al_2O_3 „*Aluminum oxide*“
  CeO_2 „*Cerium oxide*“
  $Al_2O_3/SiO_2/SiC/Si$ „*Aluminum oxide/ Silicon oxide/ Silicon carbide/ Silicon*“

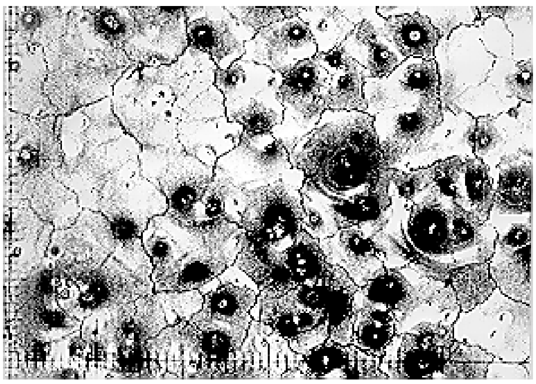
- Three layer coating system on C/SiC:
  Al_2O_3/SiO_2 as surface layer on $MoSi_2/CeO_2$ and $MoSi_2/Si$
  Al_2O_3/SiO_2 as surface layer on $MoSi_2/CeO_2$ and $MoSi_2$
  ZrO_2 as surface layer on $MoSi_2/CeO_2$ and $MoSi_2$

- Single layer coating on tungsten:
  $Zr0_2$
  Al_2O_3

4.4 Carbosil samples with single layer coating

Remelting such samples the coating usually tends to cracking and removing from the substrate regardless of the coating material. It has to be noted, however, that the original coatings already show cracks.

Remelted $Zr0_2$ (see Fig. 13) and Al_2O_3 coatings show only little cracks, and most of the coating is bonded to the substrate.



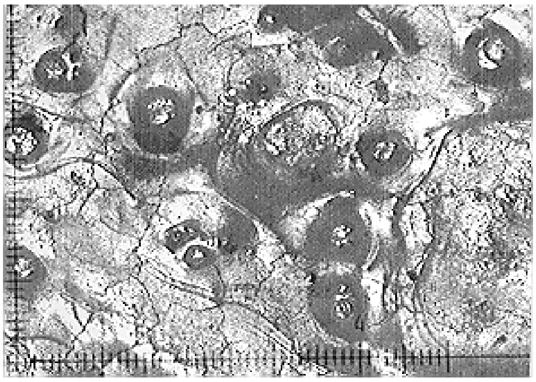
P_L=1900 W
v=21 m/min

Fig. 13: *Single layer coating: ZrO_2 on C/SiC*

$Al_2O_3/SiO_2/SiC/Si$ seems not suitable as coating material for laser remelting because it was not possible to produce overlapping tracks that did not remove from the substrate material. The coating consisting of CeO_2 shows a different behavior compared to the other materials. After the remelting process the color of the coating turns black and a „net“ of cracks develops. Using less energy there is only a gray shading of the processed track and the cracks disappear. The gray shading becomes less using less laser power. The cracks in all of the remelted coatings are caused by thermal stress while cooling down.

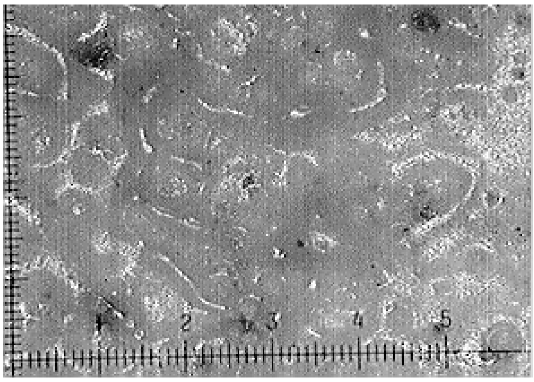
4.5 Three layer coating systems on Carbosil substrate material

The remelting of the coating systems shows good results. Here a remelting is possible without a detachment of the coating. $Zr0_2$ as surface layer shows small cracks (see Fig. 14) while the Al_2O_3/SiO_2 (see Fig. 15) surface layer is crack free.



P_L=900 W
v=10 m/min

Fig. 14: *Three layer coating system on Carbosil: ZrO_2 as surface layer on $MoSi_2/CeO_2$ and $MoSi_2$*



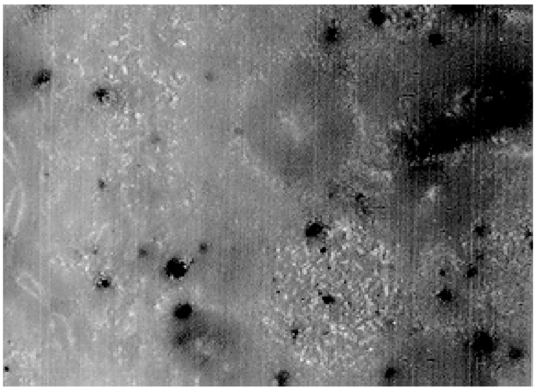
P_L=900 W
v=23 m/min

Fig. 15: *Three layer coating system on Carbosil: Al_2O_3/SiO_2 as surface layer on $MoSi_2/CeO_2$*

All of the coatings show small craters that are caused by emerging gases. During the laser remelting the gas that is in the coating because of plasma spraying assembles in bubbles which rise to the surface of the coating. Using low velocities the bubbles can emerge and craters develop. At high velocities the bubbles cannot emerge and there will be pores in the remelted coating. Suitable parameters for the remelting are a velocity of 24 m/min for Al_2O_3/SiO_2 respectively and 10 m/min for $Zr0_2$ and a laser power of 900 W.

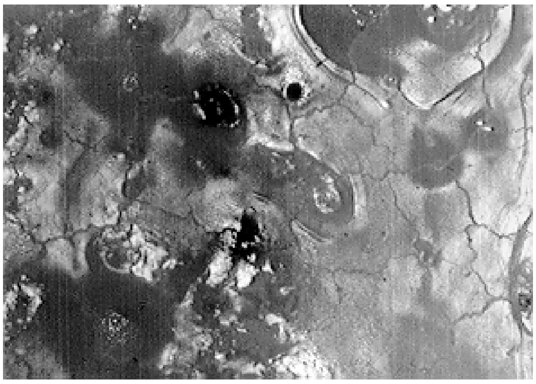
4.6 Single layer coating on tungsten

Tungsten samples with coatings of $Zr0_2$ and Al_2O_3 show good results by remelting them with suitable parameters. The laser power used was 1220 W and the velocity was 18...21 m/min for Al_2O_3 and 15 m/min for $Zr0_2$. The remelted Al_2O_3 coating (see Fig. 16) is crack free while the $Zr0_2$ coating (see Fig. 17) shows a „net“ of small cracks. Here also small craters in the surface of the coatings emerge.



P_L=1220 W
v=18 m/min

Fig. 16: *Single layer coating on tungsten: Al_2O_3 as surface layer*



P_L=1220 W
v=14 m/min

Fig. 17: *Single layer coating on tungsten: ZrO_2 as surface layer*

Detailed metallurgical investigations (X-ray scattering, EDX, scanning electron microscope, etc.) on the remelted surfaces of the plasma sprayed ceramic coatings on HT materials will be performed by DASA.

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