The role that hydrogen and sulfur play in desktop failure of thermal barrier coatings

M. Dietrich, D. Renusch, H.-E. Zschau, M. Schütze

Email: renusch@dechema.de

Funded by: Deutsche Forschungsgemeinschaft (DFG)
Part 1 Period: 01.07.2003 – 30.06.2008
Part 2 Period: 01.10.2006 – 01.10.2008

Deutsche Forschungsgemeinschaft DFG

Abstract

Under certain conditions after thermal treatment, thermal barrier coatings (TBC) show a failure mode which is called “desktop failure”. It describes the spalling of the coating after cooling, not occurring immediately but with a delay of several days. A more profound understanding of this failure mechanism can give information for the lifetime prediction of TBC. Consequently, a joint research project between the Karl Winnacker Institute an NASA has formed for the purpose of investigating this interesting subject. The NASA partner will participate in the up and coming part 2 of this project. The Result from Part 1 are presented below.

Super alloy samples coated with a plasma sprayed Yttria stabilized zirconia (YSZ) TBC were oxidized at 1100°C in 3 different atmospheres for varying dwell times. The influence of these different treatments on bondcoat depletion, TGO growth and possible desktop failure was studied. Crack growth in TBC causes acoustic emission (AE). This AE was monitored for several days after cooling the samples to room temperature. The AE was also analyzed during four point bend testing of the pre-oxidized samples.

Experimental

For the experiments CM247 samples were used with a 150 µm thick, VPS sprayed PWA 286 bond coat and a 300 µm APS coated YSZ TBC. The samples were oxidized at 1100°C for up to 1200 h in the following atmospheres: dry air, humid air with a dew point of 20°C and dry air containing 0.01 Vol.% of SO_2. After oxidation cross-sections of some samples were prepared in order to study the evolution of the thermally grown oxide (TGO). Some of the samples were connected to acoustic emission sensors just after cooling down to room temperature. Thus, the acoustic emission of the samples could be registered and analyzed. Adhesion tests on bend samples were conducted by using an established four point bend test, which should reveal the influence that water vapor and atmospheric sulfur has on the top coat adhesive strength. This test uses acoustic emission during a four point bend test at room temperature to measure both the critical strain for delamination and through cracking of APS-TBCs.

Results

Cross sections of the oxidized samples showed virtually no evidence of a difference in the thickness evolution of the TGO in the three different atmospheres. Only the TGO grown in wet atmosphere for 1200 h have less uniform characteristics (Figure 1b). However the depletion zone becomes thicker in the succession dry air, air with SO_2 and wet air (Figure 1d).

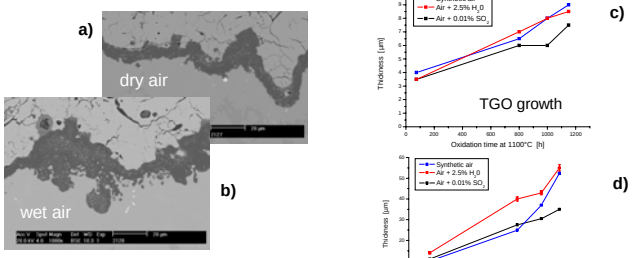

Figure 1 TGO growth
a) dry atmosphere after 1200 h at 1100°C
b) wet atmosphere after 1200 h at 1100°C
c) TGO thickness as a function of atmosphere and oxidation time
d) ß-phase depletion zone thickness as a function of atmosphere and oxidation time

After cooling the oxidized samples showed acoustic emission during more than 10 days indicating that sub-critical crack growth occurs for a long time. The acoustic emission fades away exponentially. Acoustic emission analysis during the four point bend test was able to detect two different types of damage events. A first maximum in acoustic emission during bending is attributed to delamination cracking of the TBC.

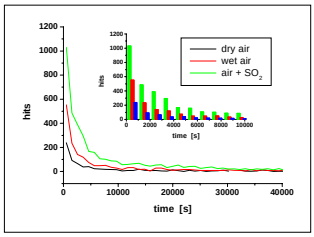

Figure 2 Acoustic emission of oxidized samples after cooling down

Later a more intense acoustic emission is correlated to through cracking of the TBC which means complete failure (Figure 3). In some experiments the delamination peak is clearly observable, in others it is not detectable. This may be explained by the fact that delamination occurs just before the through-crack so that both maxima are overlapping. An other possibility is that the sound emitted by delamination can not be detected because of a too low signal to noise ratio.

Comparison of the amount of acoustic emission did not result in clear results up to now. The number of hits and the total acoustic energy emitted shows no obvious correlation with oxidation time and atmosphere (Figure 4a). A problem in measuring acoustic energy is the difficulty to guarantee a uniform contact between wave-guides and the sound sensors leading to different attenuation. Nevertheless an interesting result shows up in comparing the critical strain to failure of the samples.

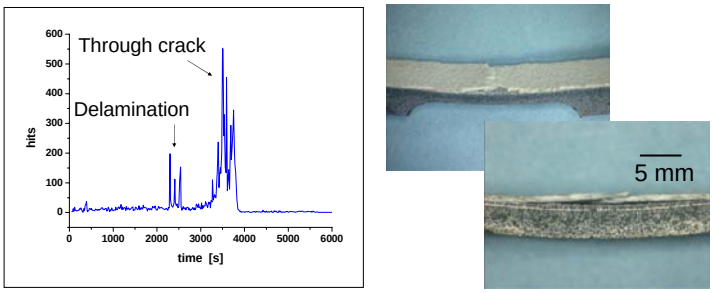

Figure 3 allowing a) Acoustic emission of oxidized samples during 4-point bend testing to distinguish between delamination and through cracking
b) Failed 4-point bend sample after the measurement

The oxidation in wet atmosphere reduces the critical strain compared to oxidation in dry air (Figure 4b). The critical strain for samples oxidized in SO_2-containing atmosphere presented an intermediate value between these two. This can be seen as first indication, that water vapor and sulfur have an influence on TBC-failure.

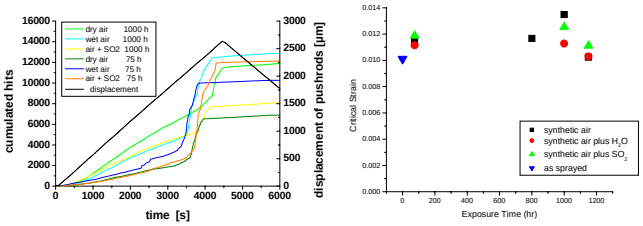

Figure 4 a) Cumulated acoustic emission during the 4-point bend test
b) Critical strain as a function of oxidation conditions compared to the as sprayed state

The presence of hydrogen in the TGO region between top coat and bond coat is supposed to play an important role for the occurrence of TBC top coat failure. Therefore the detection of hydrogen with concentrations in the ppma region in the TGO is an important part of the project. Unique methods to detect hydrogen in the ppm-region with spatial and depth resolution are particle induced gamma emission (PIGE) and nuclear reaction analysis (NRA) belonging to the ensemble of ion beam analysis (IBA). However, hydrogen from laboratory air may contaminate the sample after high temperature exposure and measures have to be taken to prevent this pollution. Both approaches were followed in this project.

For PIGE measurements the TBC was removed from the sample just before measurement in a new developed vacuum experimentation chamber (Figure 5a) containing a simplified 4-point bend mechanism (Figure 5 b). It was shown that with this device the coating can be spalled reliably from the sample surface.

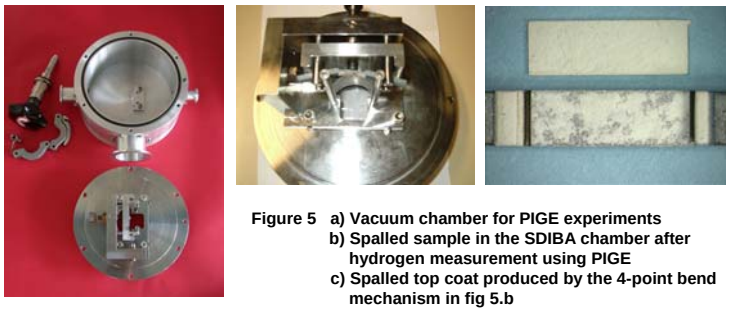

Figure 5 a) Vacuum chamber for PIGE experiments
b) Spalled sample in the SDIBA chamber after hydrogen measurement using PIGE
c) Spalled top coat produced by the 4-point bend mechanism in fig 5.b

Results from H-concentration measurements in the TBC systems after different oxidation times reveal a drying effect with increasing exposure time in dry air (Figure 6), i.e. some moisture must have come into the system before high temperature exposure.

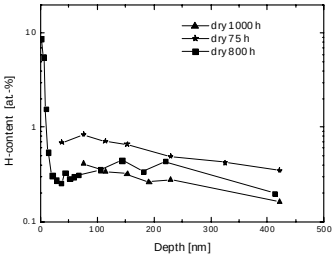

Figure 6 H-depth profiles of samples oxidized in dry atmosphere for 75, 800 and 1000 hours