RAMAN STUDIES ON EB-PVD 7 wt% YTTRIA-STABILIZED ZIRCONIA COATINGS WITH CMAS DEPOSITS

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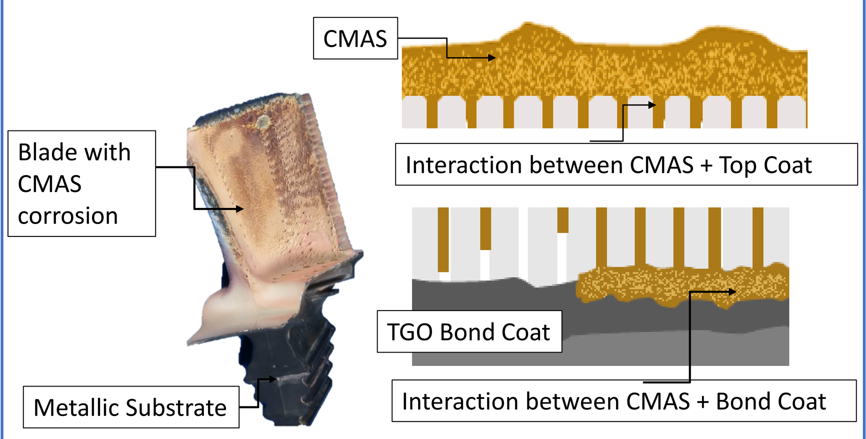
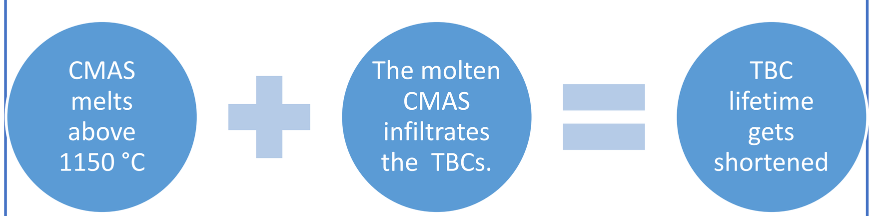
Key Words: Thermal barrier coatings (TBCs); Calcium–magnesium–alumina–silicate (CMAS); Microstructure; Raman Spectroscopy

Motivation

- EB-PVD TBCs have been used to extend the life of jet engine turbine blades by protecting the substrate alloy from extreme thermal loads.
- EB-PVD TBCs have a strain-tolerant columnar microstructure with gaps that can be filled with CMAS oxides.
- Calcium-magnesium-aluminum-silicate (CMAS) or CaO, MgO, Al_2O_3, SiO_2, can deteriorate TBCs when this sand like material is ingested in the turbine.

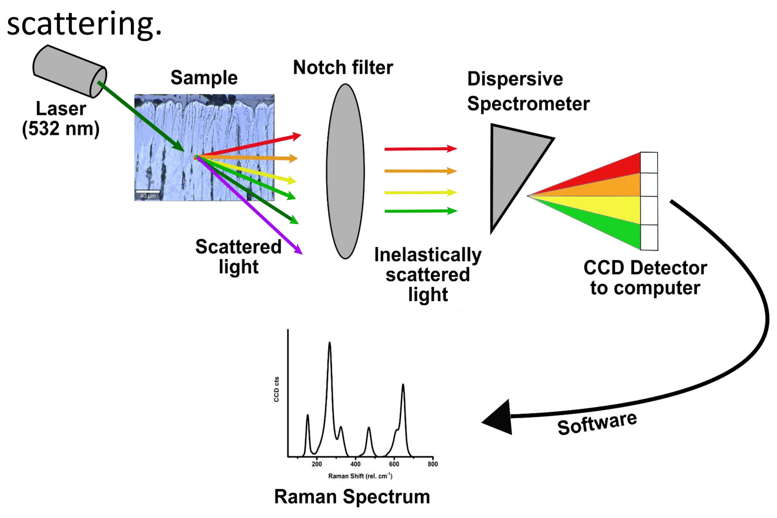




- CMAS affects the life of TBCs due to thermochemical attack and thermomechanical effects
- Knowing the impact of CMAS interaction will help to quantify and mitigate their effects
- Raman spectroscopy has the potential to track phase changes from CMAS ingestion

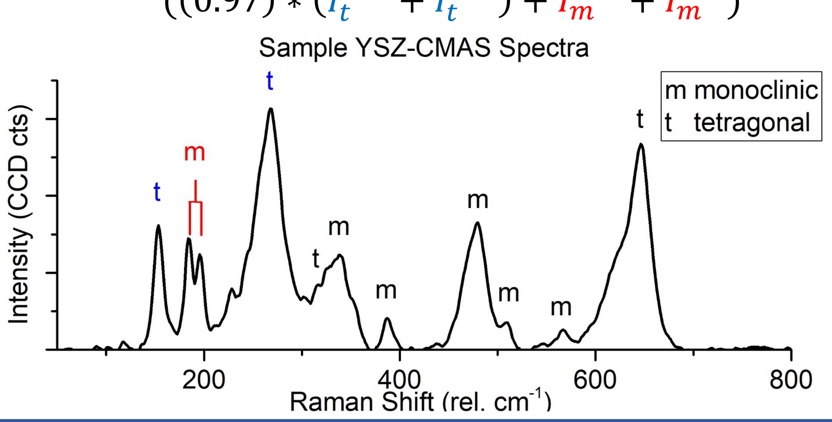
Methods

- Raman spectroscopy is a nondestructive technique used to investigate the composition of materials scattering.

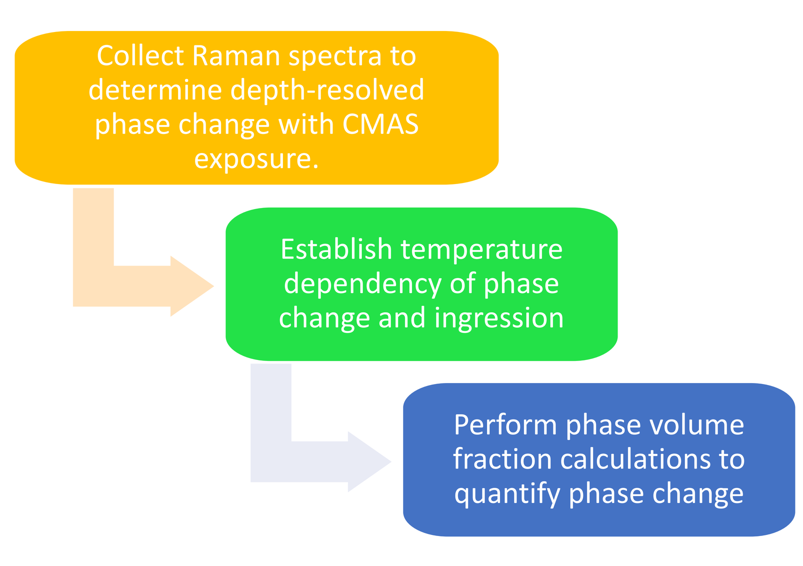


- The unique spectra caused by vibrational modes of bonds in the material can be linked to specific compositions: a chemical fingerprint
- Monoclinic Phase volume fraction can be calculated

$$f_m = \frac{(I_m^{182} + I_m^{191})}{((0.97) * (I_t^{148} + I_t^{263}) + I_m^{182} + I_m^{191})}$$



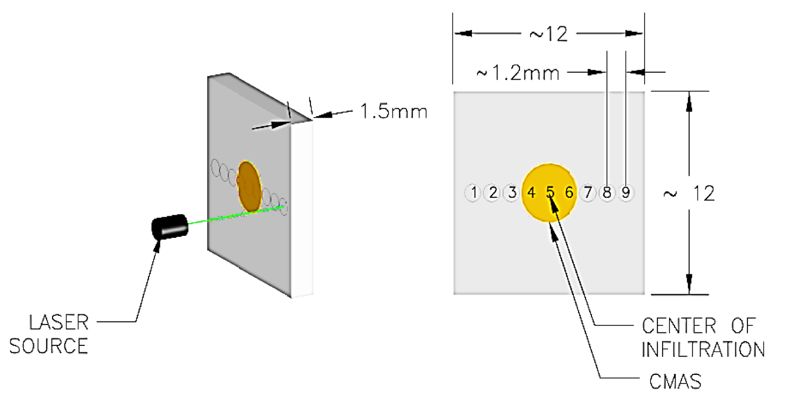
Objectives



Experiment Setup

- 7YSZ was deposited on alumina substrates using EB-PVD
- CMAS is deposited in the center of the sample

label	Composition	Temperature (°C)	Total time (h)
B1	7YSZ+CMAS 1	1225	10
B2	7YSZ+CMAS 1	1250	1
B3	7YSZ+CMAS 1	1250	10



- Points of interest: Area outside CMAS infiltration (3), edge of CMAS infiltration (4) and CMAS center of infiltration (5).

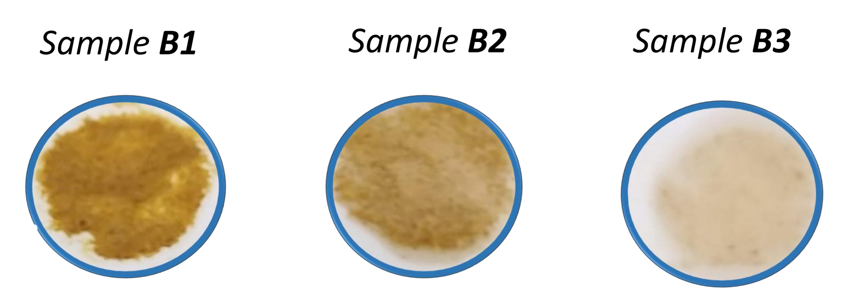
Raman Spectra Collection Parameters

- 300RA WITec Confocal Raman microscope
- He:Ne 532 nm laser source
- Laser power: 22mW

CMAS 1 Composition (Melting Temperature 1230°C)

Oxide	CaO	MgO	Al_2O_3	SiO_2	FeO	TiO_2
Wt%	22	8	18	40	10	2

Initial Observations



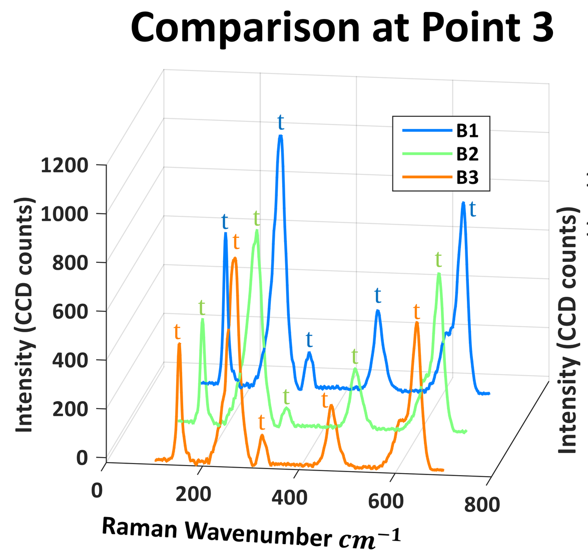
Sample B1, B2 and B3 showing the deposits after infiltration and heat treatment.

Preliminary qualitative analysis using an optical microscope at center point of infiltration (point 5):

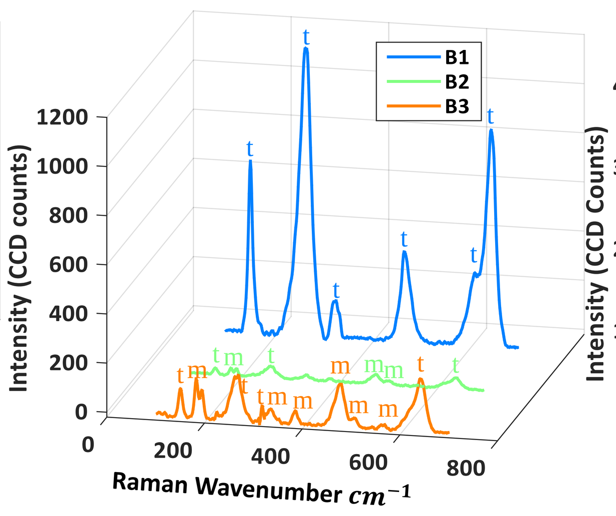
- CMAS begins to melt, react, and infiltrate between 1230°C and 1250°C.
- B1 displays distinctive separation between CMAS grains and TBC surface: No indication of CMAS interaction with 7YSZ.
- Less CMAS was found on top of samples B2 and B3 compared to B1

Results as a Function of Location & Heat Treatment

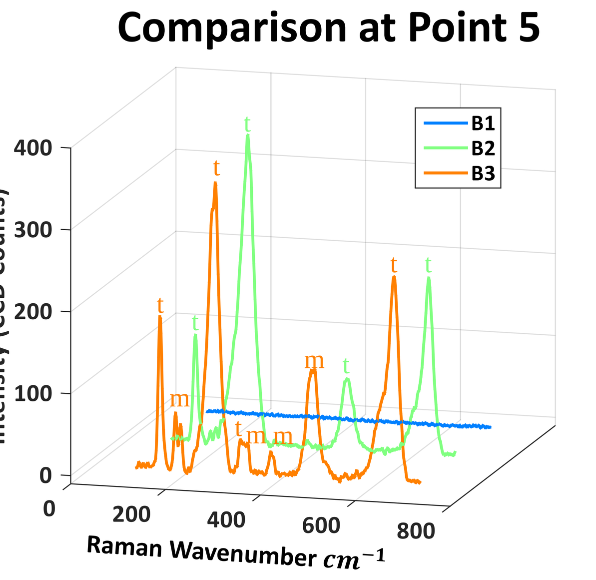
Raman Spectra Comparison at Point 3



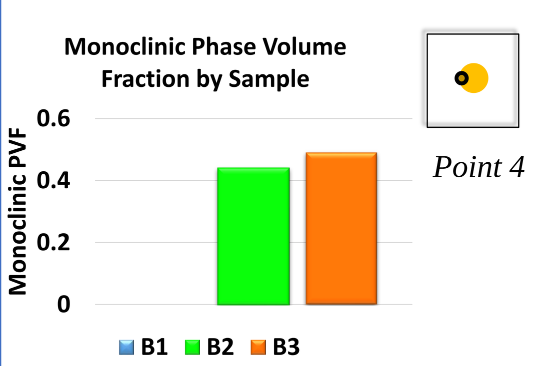
Raman Spectra Comparison at Point 4

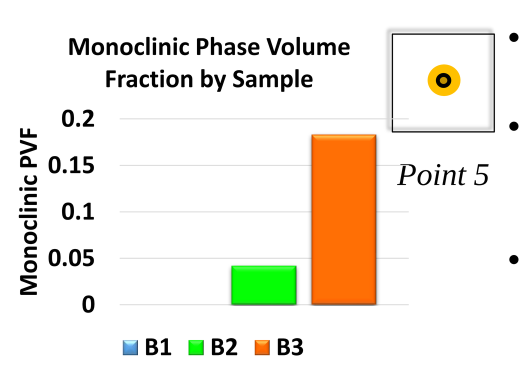


Raman Spectra Comparison at Point 5



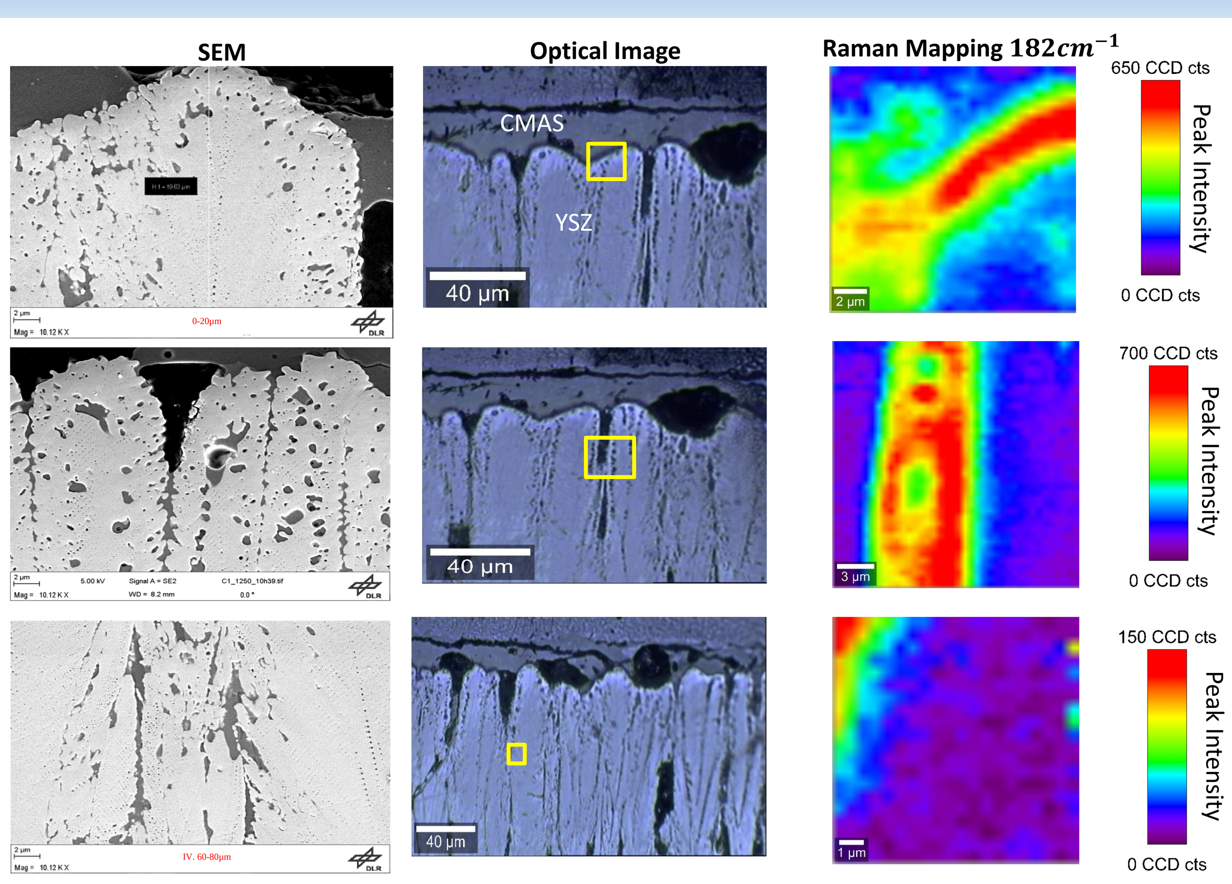
- At point 3 there was no monoclinic phase signal found for any sample
- Monoclinic peaks were found for both B2 and B3 at CMAS-localized areas indicating a reaction has taken place
- B3 has the most dominant monoclinic peaks throughout as a result of longer heat treatment
- Point 5, shows less intense signals compared to point 4 as a result of the thick CMAS overlay





- B3 has PVF of 0.5 and 0.18 for points 4 and 5 respectively
- For both B2 and B3, point 4 shows greater monoclinic PVF indicating more desolation of the coating in this region
- Point 5 has reduced signal for all samples due to the CMAS overlay

2D Raman Imaging of B3 (1250°C, 10h)

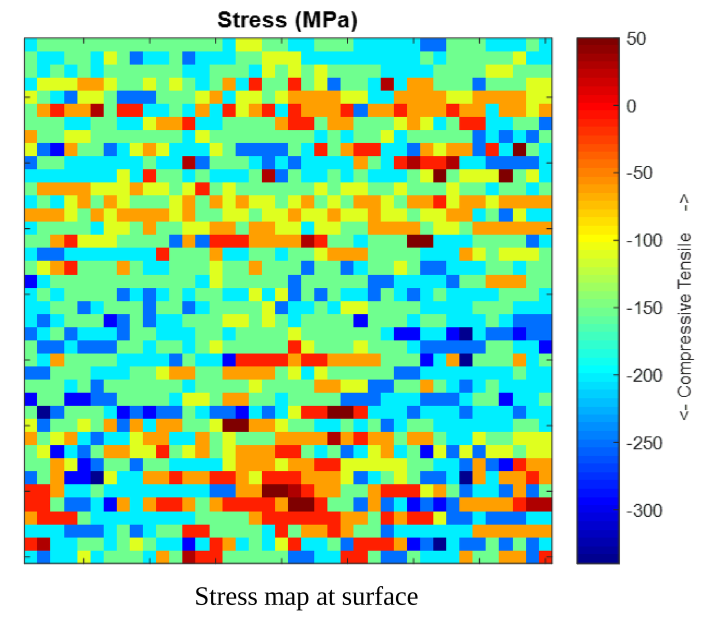


Images from upcoming paper by Bohorquez et al.

- 7YSZ coating is most susceptible to monoclinic phase formation in the regions where the coating is in direct contact with the molten CMAS
- Monoclinic phase is reduced as depth into sample increases
- Presence of tetragonal is represented by areas with low intensity
- Raman can distinguish between monoclinic and tetragonal phases
- Raman mapping can substitute phase identification techniques such as TEX, XRD

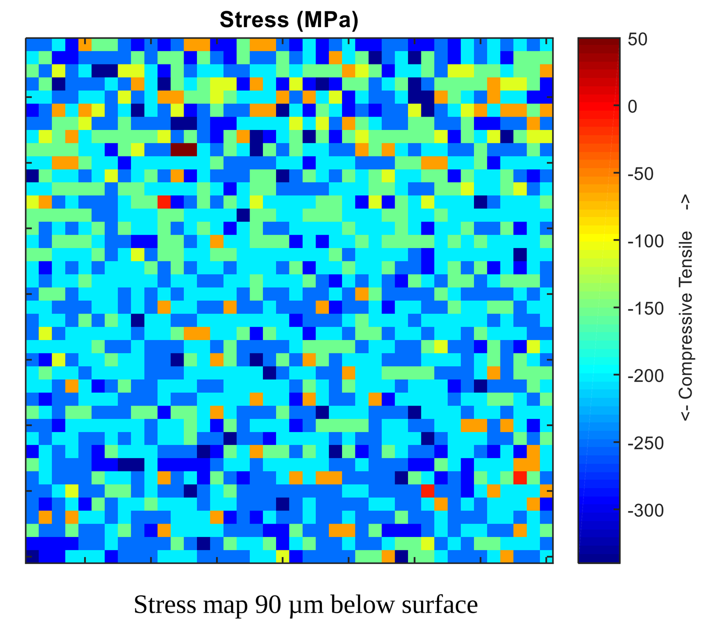
Stress Measurements on B3

- Tracking the shift of the 640 cm^{-1} Raman peak allows us to know about the type of stresses that are present within our TBC



Stress map at surface

- At the surface of the coating where there is more of the monoclinic phase, the stress map shows more tensile stress



Stress map 90 µm below surface

- Raman spectroscopy can also give us depth resolved measurements
- At a depth of 90 µm where the monoclinic phase is in smaller quantities, the stress is here is more compressive

Conclusions

- These results show that monoclinic peak identification is correlated with CMAS infiltration.
- Monoclinic phase volume fraction directly depends on CMAS infiltration temperature and reaction time.
- Raman imaging appears as an effective technique, not requiring any specific surface preparation, to track the 182 peak distribution in zirconia scales after oxidation due to CMAS.
- The method provides, in a single scan, not only structural and mechanical information, but also information about the corrosion process.
- Raman Imaging appears ass an effective technique, not requiring any specific surface preparation, to track the 182 cm^{-1} peak distribution in zirconia scales after oxidation due to CMAS

Acknowledgments

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Reference:
Bohorquez E., Sarley B., Hernandez J., Hoover R., Tetard L., Naraparaju R., Schulz U., Raghavan S., *Investigation of the Effects of CMAS-infiltration in EB-PVD 7% Yttria-Stabilized Zirconia via Raman Spectroscopy.* Extended abstract accepted for the 59th AIAA/ASCE/AHS/ASC Structural Dynamics, and Materials Conference, AIAA SciTech Forum, 8-12 January 2018, Orlando, FL.