Synchrotron X-Ray Diffraction Study of CMAS Ingression in Electron-Beam Physical Vapor Deposition Thermal Barrier Coatings

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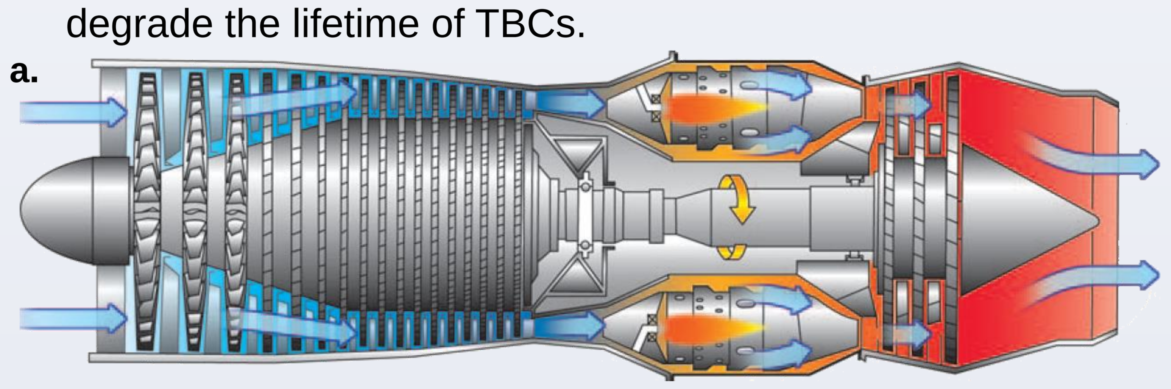
Background and Introduction

- Yttria-stabilized zirconia (YSZ) is a ceramic topcoat for thermal barrier coatings (TBCs) in gas turbines.
- TBCs protect turbine blades from extreme operating temperatures.
- Deposits, such as sand (Calcium-magnesium-aluminum-silicate CMAS) or volcanic ash, become molten, infiltrate and degrade the lifetime of TBCs.

a.



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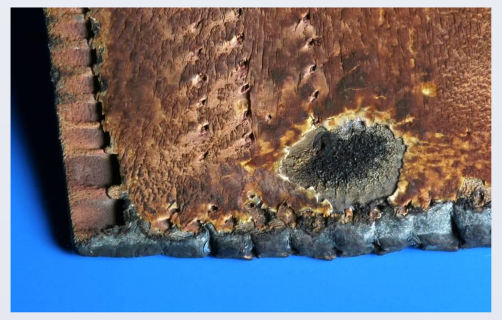


Images of turbine blades have been used with permission by the German Aerospace Center (DLR)

a. Full depiction of an jet engine.
b. A full image of a high power turbine blade exposed to CMAS during operation.
c. A close-up of a high power turbine blade that has been exposed to CMAS during operation.

Motivation and Objectives

Motivation:

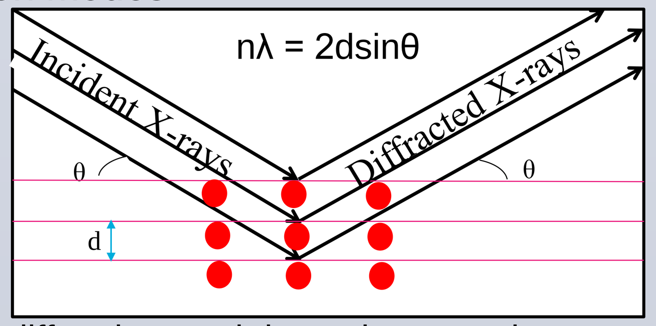
- 2D X-ray Diffraction (XRD) provides high-resolution biaxial strain data for all phases present, allowing for a better understanding of how the introduction and ingression of CMAS degrades the lifetime of thermal barrier coatings.

Objectives:

- Obtain in-plane (e11, e22) strain data at room temperature.
- Observe how CMAS has accelerated phase transformations within the coating and how these transformations have influenced the strain in the coating.

Theory

- XRD can provide information about the crystal structure, phase, and strain when the X-ray beam interacts with the material of interest.
- The angle of diffraction relates to the spacing of the atomic planes by Bragg's Law (Figure below):
  $n\lambda = 2d\sin\theta$
- XRD experiments can be performed in reflection or transmission modes



X-ray diffraction and how the rays interact with crystal planes within a sample where n = integer values, λ = wavelength of X-ray, and d = d-spacing

Methods and Materials

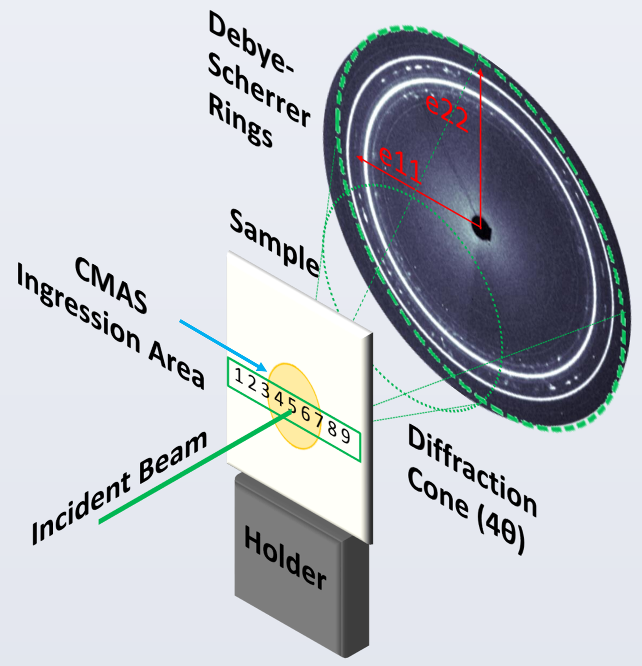
- A high-energy (71 keV) X-ray beam of 30 x 300 μm^2 size impinged on samples.
- With the geometry used, diffraction information is collected roughly in two directions normal to the incident beam in the form of Debye-Scherrer rings.
- Shape and sizes of the rings change depending on the presence of internal strains.
- Phase and strain in the material can be determined referencing to known d-spacing from XRD databases.

Label	Composition	Temperature (°C)	Total Time (h)
A0	Pure 7YSZ	0	0
B3	7YSZ+CMAS	1250	10

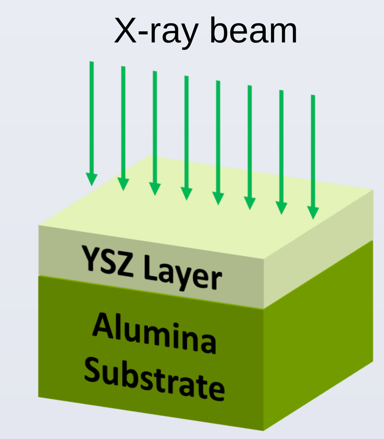
Samples used during this study

CMAS Chemical Composition						
Elements	SiO_2	CaO	Al_2O_3	FeO	MgO	TiO_2
Wt %	40	22	18	10	8	2

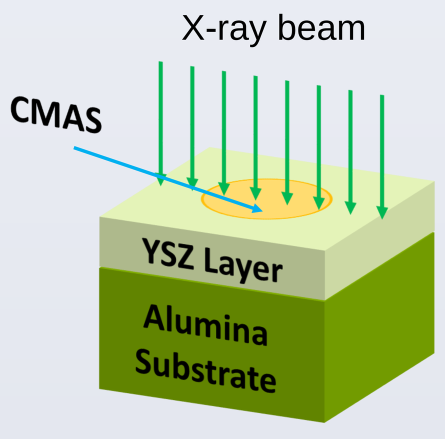
The chemical composition of CMAS by weight percentage



Schematic view of XRD data collection

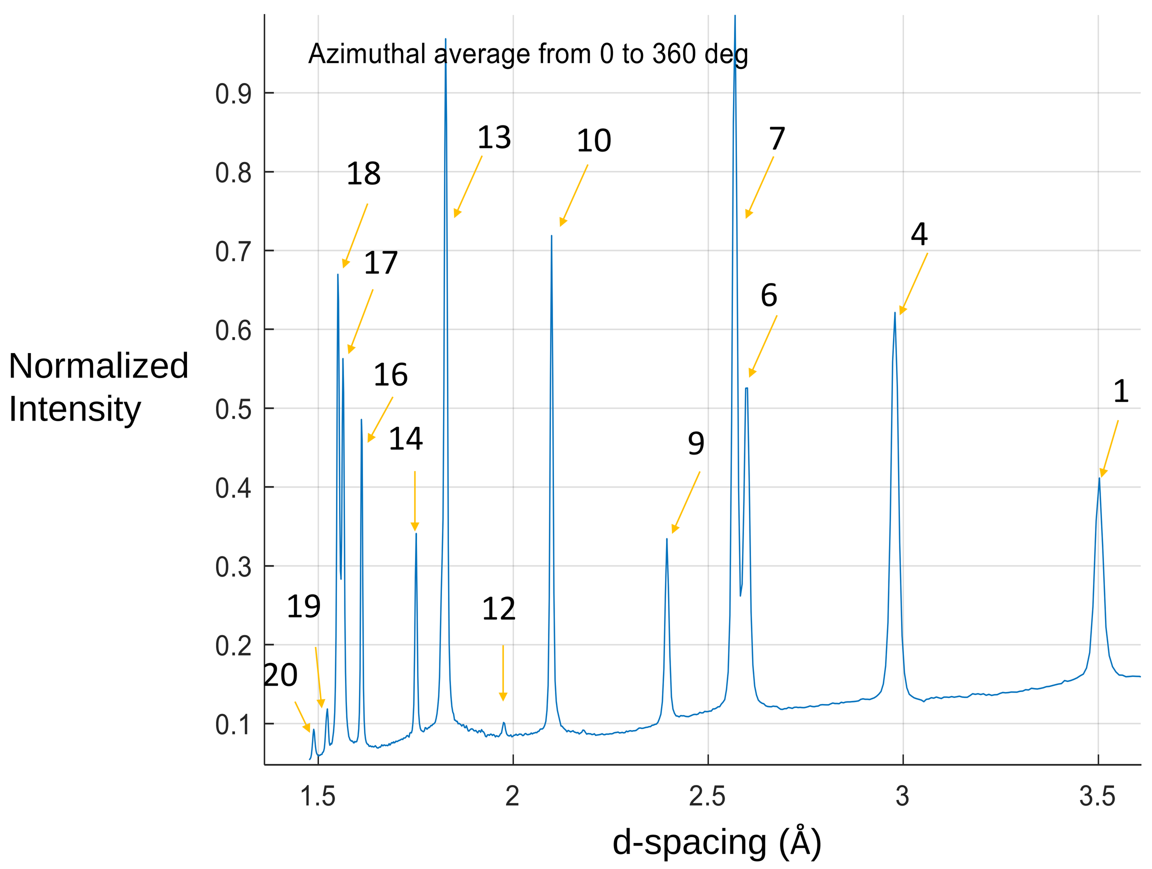


Sample without CMAS

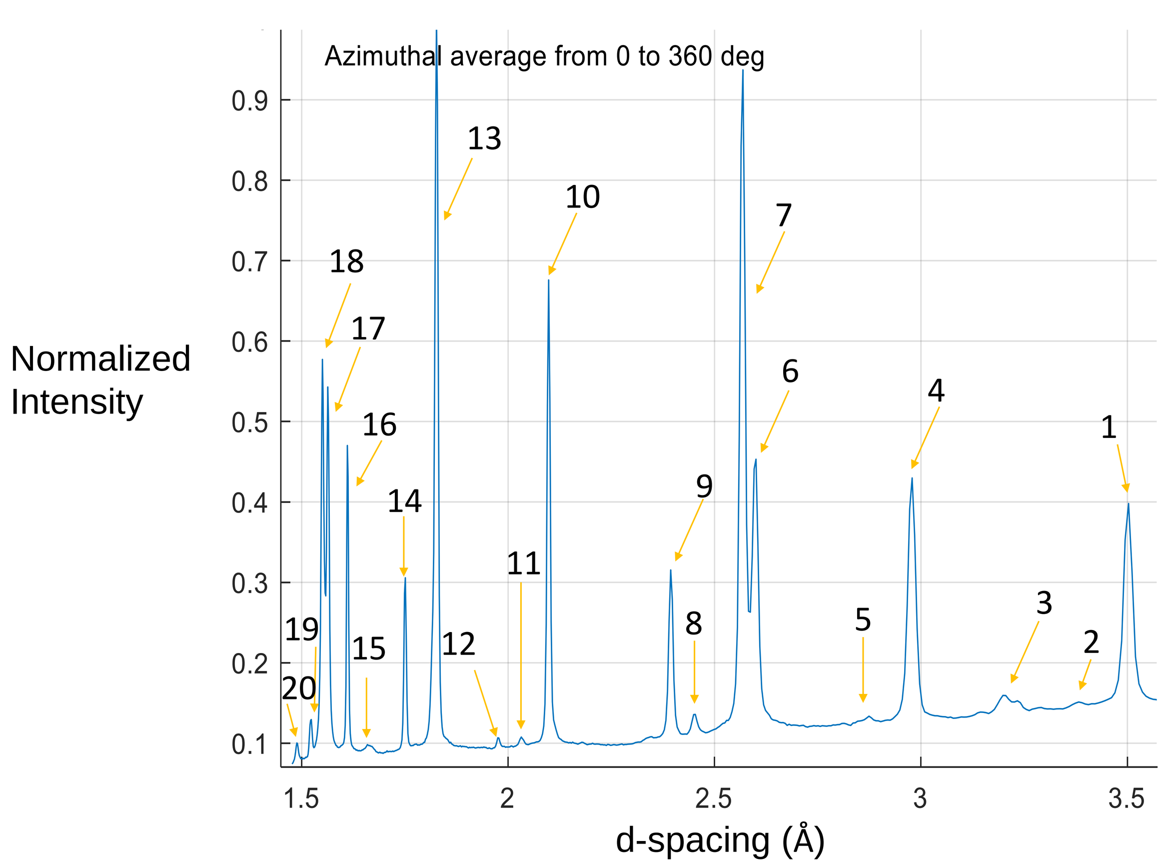


Sample with CMAS

Peak Identification



Intensity vs d-spacing of sample without CMAS



Intensity vs d-spacing of sample with CMAS

Peak	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Material	Al_2O_3	CMAS SiO_2	YSZ (m)	YSZ (t)	YSZ (m)	YSZ (t)	Al_2O_3	CMAS SiO_2	Al_2O_3	Al_2O_3	CMAS Al_2O_3 (o)	Al_2O_3	YSZ (t)	Al_2O_3	CMAS SiO_2	Al_2O_3	YSZ (t)	YSZ (t)	Al_2O_3	YSZ (t)

Identification of materials and phases in the samples with and without CMAS from the XRD peaks shown above

Summary

- High-resolution XRD experiments have been performed using coated samples with and without CMAS at Argonne National Laboratory.
- The peaks of YSZ and alumina were obtained from the XRD data of the sample without CMAS.
- The sample with CMAS provides the same peaks as the sample without CMAS, though less intense because of the ingression of CMAS into the YSZ coating
- Additional peaks have been observed due to the presence of CMAS.

Future Work

Next Steps of this Study

- Determining of the amount of tetragonal and monoclinic YSZ phases will provide better understanding of the effect of CMAS ingression in the coating.
- Calculation of strain will enable to understand the effects of CMAS ingression on residual strain in the coating.

In-situ XRD with CMAS infiltration

- Use of in-situ XRD to observe infiltration of CMAS into the coating as it occurs during a full thermal cycle.
- This study would provide strain components, phase composition, and CMAS infiltration over time into the coating.
- In addition, this study could use different deposition techniques of applying TBCs and compare their resistance to CMAS.

CMAS ingression of Environmental Barrier Coatings

- Environmental Barrier Coatings (EBCs) allow for higher temperatures than TBCs can withstand.
- These coatings are more susceptible to CMAS infiltration due to higher operating temperatures.
- The current study can be extended to investigate the CMAS ingression in EBCs.

References/Acknowledgements

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