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

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Background and Introduction

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

Motivation and Objectives

\textbf{Motivation:}

\begin{itemize}
  \item 2D X-ray Diffraction (XRD) provides high-resolution bi-axial strain data for all phases present, allowing for a better understanding of how the introduction and ingestion of CMAS degrades the lifetime of thermal barrier coatings.
\end{itemize}

\textbf{Objectives:}

\begin{itemize}
  \item Obtain in-plane (e11, e22) strain data at room temperature.
  \item Observe how CMAS has aggregated phase transformations within the coating and how these transformations have influenced the strain in the coating.
\end{itemize}

Theory

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

Methods and Materials

\textbf{Label} | \textbf{Composition} | \textbf{Temperature (°C)} | \textbf{Total Time (h)}
\hline
A0 & Pure YSZ & 0 & 0
B3 & YSZ+CMAS & 1250 & 10
\hline
\textit{Samples used during this study}

\textbf{CMAS Chemical Composition}

\begin{tabular}{|l|l|l|l|l|l|l|}
\hline
Element & SiO\textsubscript{2} & CaO & Al\textsubscript{2}O\textsubscript{3} & FeO & MgO & TiO\textsubscript{2} \\
Weight % & 40 & 22 & 18 & 10 & 8 & 2 \\
\hline
\end{tabular}

This chemical composition of CMAS by weight percentage

\textbf{Peak Identification}

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

Summary

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

Future Work

\begin{itemize}
  \item Determining the amount of tetragonal and monoclinic YSZ phases will provide better understanding of the effect of CMAS ingestion in the coating.
  \item Calculation of strain will enable to understand the effects of CMAS ingestion on residual strain in the coating.
  \item Use of in-situ XRD to observe infiltration of CMAS into the coating as it occurs during a full thermal cycle.
  \item This study would provide strain components, phase composition, and CMAS infiltration over time into the coating.
  \item In addition, this study could use different deposition techniques of applying TBCs and compare their resistance to CMAS.
\end{itemize}

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