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Crystallographic Polymorphs Changes of the Yttria Stabilized Zirconia under Humidity

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Abstract

One of the major sources of efficiency loss in gas turbine engines is the leakage of high-pressure gases through the clearance between rotating blade tips and the stationary shroud. To minimize this leakage in the high-temperature turbine section, yttria-stabilized zirconia (YSZ)-based abradable coatings are commonly applied by plasma spraying. These coatings provide a self-adjusting sealing system that enables minimal tip clearance during engine operation. In this study, a commercial YSZ-based abradable coating (Metco 2460NS) was exposed to a humid environment at 60˚C for 85 weeks, after which a decrease in hardness was observed. The phase composition before and after exposure was characterized by X-ray diffraction (XRD) coupled with Rietveld refinement. The results revealed significant phase evolution during aging, accompanied by a progressive reduction in mechanical bond strength. The observed mechanical performance degradation was associated with phase transformations within the YSZ coating. The stabilization of zirconia by yttrium is primarily attributed to the formation of oxygen vacancies in the crystal lattice. Prolonged exposure to moisture may alter the defect chemistry and promote local yttrium redistribution or depletion, thereby destabilizing the cubic and tetragonal phases and mainly, increasing the fraction of the monoclinic phase. The associated volume expansion of the monoclinic phase can generate localized stresses and microcracking, ultimately compromising coating integrity. These findings suggest that prolonged hydrothermal exposure promotes phase destabilization and contributes to the deterioration of the mechanical performance of YSZ-based abradable coatings.

Keywords

Yttria-Stabilized Zirconia, Abradable Coating, Hydrothermal Aging, Phase Transformation, Rietveld Refinement

1. Introduction

A gas turbine engine extracts energy from the hot, high-pressure gases generated during fuel combustion. Ideally, the maximum possible amount of this energy is converted into useful mechanical work by the turbine to drive compressors, generators, or propellers, thereby producing power or thrust. Any energy remaining in the exhaust stream represents unrecovered energy and reduces the overall engine efficiency. One of the primary sources of efficiency loss is the leakage of high-pressure gases through the clearance between the rotating blade tips and the stationary shroud. This bypass flow does not contribute to torque generation and therefore decreases the amount of useful work extracted by the turbine. To minimize tip leakage, significant efforts have been devoted to reducing blade tip clearance through the application of abradable coatings on the turbine shroud [1]. These coatings allow controlled blade incursion during operation, enabling the blade tips to establish a minimum running clearance without causing blade damage. Consequently, gas leakage is reduced while safe operation is maintained. The efficiency of gas turbine engines is therefore strongly dependent on the blade tip-to-shroud clearance, with smaller clearances resulting in lower leakage losses, higher energy extraction, and improved overall efficiency. Considerable research has been devoted to identifying suitable ceramic materials for abradable coating applications. Among the most widely used materials is yttria-stabilized zirconia (YSZ), owing to its excellent high-temperature stability. Numerous studies have investigated the incorporation of additional stabilizing oxides to further improve the thermal stability and durability of YSZ under severe operating conditions, including those encountered in gas turbine engines [2]-[5]. YSZ abradable coatings are typically deposited by plasma spraying, a process in which feedstock particles are exposed to temperatures exceeding 2500˚C [6]. To produce a porous and interconnected microstructure with enhanced abradability, the feedstock powder is blended with a polymeric pore-forming agent. During spraying and subsequent thermal exposure, the polymer burns out, creating an interconnected pore network that improves the coating's abradable characteristics. The addition of yttrium oxide to zirconia (ZrO_2) does not impart water-repellent properties. Rather, yttrium stabilizes the zirconia crystal structure, modifies the surface energy, and influences the interaction of the material with moisture [7]. Specifically, yttrium stabilizes the tetragonal and/or cubic zirconia phases at room temperature, suppressing the phase transformations that can otherwise lead to cracking. The substitution of Y^{3+} ions for Zr^{4+} ions also generate oxygen vacancies within the zirconia lattice, which facilitate the adsorption and diffusion of water molecules and ionic species. As a result, YSZ surfaces are generally hydrophilic and readily form surface hydroxyl groups in humid environments. In the present work, the degradation of the mechanical properties of a YSZ abradable coating exposed to a high-humidity environment at 60˚C for 85 weeks is investigated. The degradation is attributed to changes in the relative phase composition of the yttria-stabilized zirconia and to an increase in the monoclinic phase fraction. These changes were

evaluated by comparing the Rietveld-refined X-ray diffraction (XRD) patterns of the coating before and after prolonged moisture exposure [8]. The results demonstrate a clear correlation between hydrothermal aging, phase evolution, and the deterioration of the coating's mechanical performance.

2. Experimental

Samples of abradable coatings with a thickness of 3 - 4 mm, manufactured from Metco 2460NS in accordance with commercial specifications and processing requirements, were prepared for this study. The chemical composition of the Metco 2460NS weight percent includes a balance of 7.5% Y_2O_3, non-definite binder and 4% polymer (burned in the process) in the ZrO_2 phase. The thermal plasma spraying process exceed 2500˚C and above. METCO 2460NS powder was mixed with argon gas and deposited onto the samples at a gun traverse speed of 1000 mm/s, approximately. Tens of Ni-based superalloy (IN939) discs, each measuring 25 mm in diameter and 3 mm in thickness, were prepared to achieve abradable coating thicknesses of 3 - 4 mm for reference samples and long-term exposure samples in a humidity cell. The post-processing heat treatment consisted of heating the samples from room temperature to 450˚C at a rate of 5˚C - 8˚C/min, followed by an 8-hour hold at 450˚C [9]. Representative specimens were characterized before the experiment for comparison, and after the environmental exposure. Removal intervals after environmental exposure change between weeks to months. The characterization included metallographic evaluation of porosity and tensile testing of adhesive strength in accordance with the relevant standards and commercial specifications [10]. Most of the specimens were subsequently placed in a humidity chamber maintained at a constant temperature of 60˚C. During the exposure period, the relative humidity was alternated weekly between 40% and 70%, for a total duration of 85 weeks at 60˚C. One specimen was removed after different period for tensile bond strength evaluation [10]. At the end of the exposure period, both the unexposed reference specimen and a specimen exposed for 85 weeks were examined by X-ray diffraction (XRD) at several locations across the coated discs. The measurements were performed using a Panalytical Empyrean multipurpose diffractometer equipped with a Cu Kα radiation source and a real-time multistrip detector (RTMS). The operating conditions were a tube voltage of 40 kV and a filament current of 30 mA. Diffraction patterns were recorded over a 2θ range of 5˚ - 120˚ using continuous scanning with a step size of 0.03˚. The measurement time per step was 50 s. Rietveld refinement of the XRD data revealed changes in the yttria-stabilized zirconia (YSZ) phase composition, as well as variations in the relative fractions of the crystallographic phases. The Rietveld refinement calculation was done with two different programs; Powder Cell for Windows (PCW) [11] which is a convenient tool for initial refinement and works well with crystallized fine powders with separated multiphase systems, and, the Fullprof [12] with sub program Winplotr, as a professional program supported by the European union and updated every year. PCW is simple software with limited options suitable for initial evaluation. It works very well for ideal powders. The advantage of this soft-

ware is that it is user-friendly. It is recommended to obtain initial results and qualitative analysis with some quantitative evaluation data. FullProf is professional software, works well with most polycrystalline samples, excellent to obtain precise quantitative analysis. However, it needs a lot of training to use it. Usually, we start with PCW to obtain the initial results. But the final ones are coming from the FullProf program. In case of fine powders there are good agreements between the refined parameters in both programs, but in solid sample the PCW may show different quantitative data, and it is preferred to trust on the results from FullProf. It should be noted that the results from both programs showed the same trend (increasing the monoclinic polymorph in the sample that spent long time in wet atmosphere). These phase changes were found to correlate with the degradation of the coating's bond strength following prolonged exposure to a humid environment. The bond strength was measured in Instron Model No. 5982 with a build in load cell with an accuracy of 0.15%. The cross-section load of the bond was applied on 25 ± 0.1 mm diameter sample. The repeatability and fluctuation of the bond straight measurements for the abradable coating was in the range of ±0.5 MPa.

3. Results

Figure 1 and **Figure 2** show the X-ray diffraction (XRD) patterns of the yttria-stabilized zirconia coating before and after exposure calculated with PCW and FullProf programs. The phase fractions shown in the internal box in the plot (tetragonal, monoclinic, and cubic) were determined by Rietveld refinement and correspond to the coating before and after high-moisture exposure, respectively [8]. In **Figure 3** a representative histogram of the phase quantitative to emphasize the change in the phases fraction, before (red) and after exposed for 85 weeks (blue). The histogram shows the increase of the monoclinic phase, the formation of the tetragonal phase and a decrease of the cubic phase during the exposure. The same patterns were calculated by the Fullprof program which reveals that the cubic phase is a pseudo-cubic phase when the a/c lattice ratio is 1.01 - 1.02, practically not a real cubic phase but tetragonal. In this case, the cubic phase and the tetragonal phase represented in **Figure 3** can be united as a pseudo cubic phase when the total amount is 81.64% ZrO_2 cubic phase and 18.36% monoclinic phase before exposure to moisture, and 76.33% ZrO_2 tetragonal phase and 23.67% monoclinic phase after the exposure to moisture. Simultaneously, the bond strength degraded during the same period with some fluctuations, presented in **Figure 4**.

The bond strength was measured using a uniaxial tensile test, with the coating-oriented perpendicular to the tensile load according to the standard and as described in the experimental above [10]. **Figure 5** presents the heating cycle used to verify the stability of the phases previously identified by XRD in the sample exposed to alternate weekly humidity of 40% and 70% (RH), at 60˚C for 85 weeks, under different temperatures and aging conditions. The sample was heated under degassing conditions, and XRD patterns were collected at four stages for stability verification: at 25˚C before heating, after heating to 450˚C, after holding at 450˚C

for 30 min, and after cooling back to 25˚C. The corresponding stages are indicated in Figure 5 according to the XRD patterns. Rietveld refinement revealed no detectable changes in phase composition throughout the heating cycle.

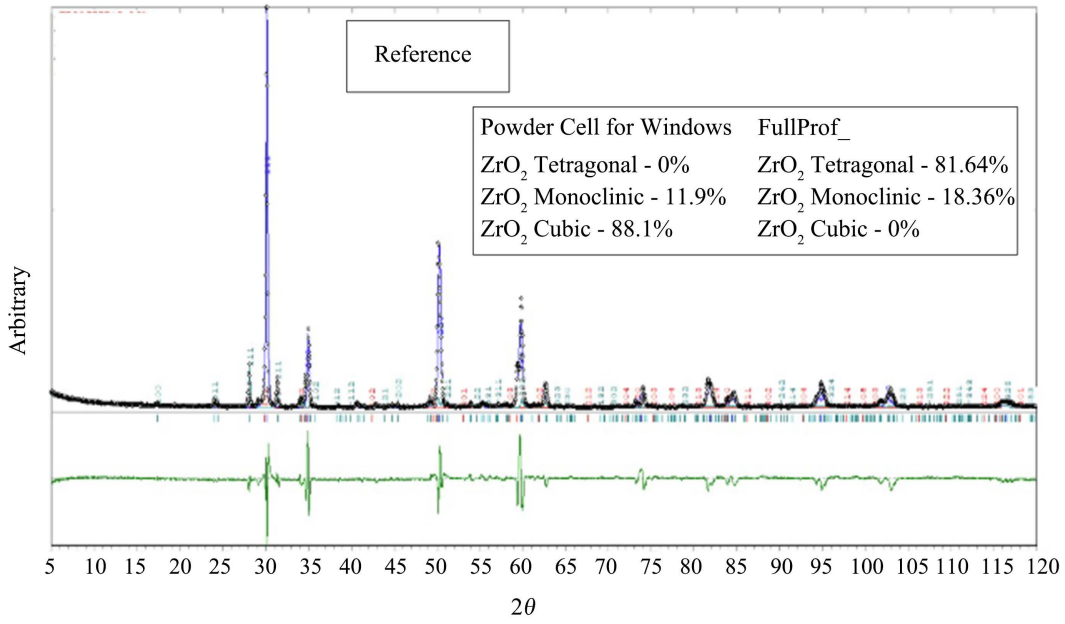


Figure 1. X-ray diffraction (XRD) pattern of yttria-stabilized zirconia (YSZ) before moisture exposure. The green line at the bottom represents the difference between the experimental diffraction pattern and the calculated fit. The insert box shows the tetragonal, monoclinic, and cubic phase fractions determined by Rietveld refinement using PCW and FullProf software.

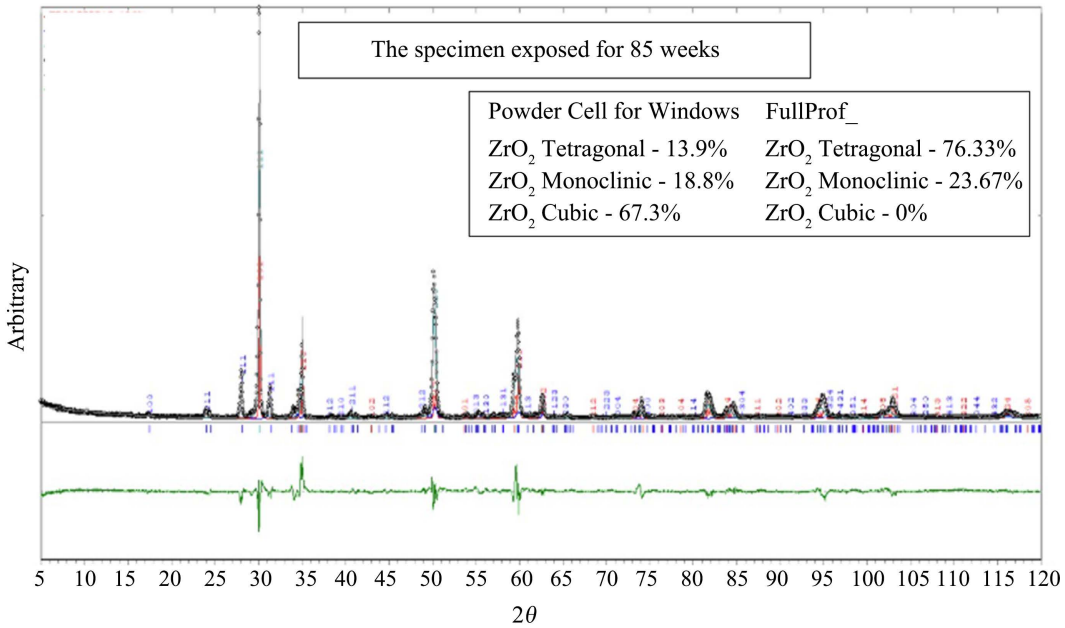


Figure 2. X-ray diffraction (XRD) pattern of yttria-stabilized zirconia (YSZ) following moisture exposure. The green line at the bottom represents the difference between the experimental diffraction pattern and the calculated pattern obtained by Rietveld refinement. The insert box shows the tetragonal, monoclinic, and cubic phase fractions determined by Rietveld refinement using PCW and FullProf programs.

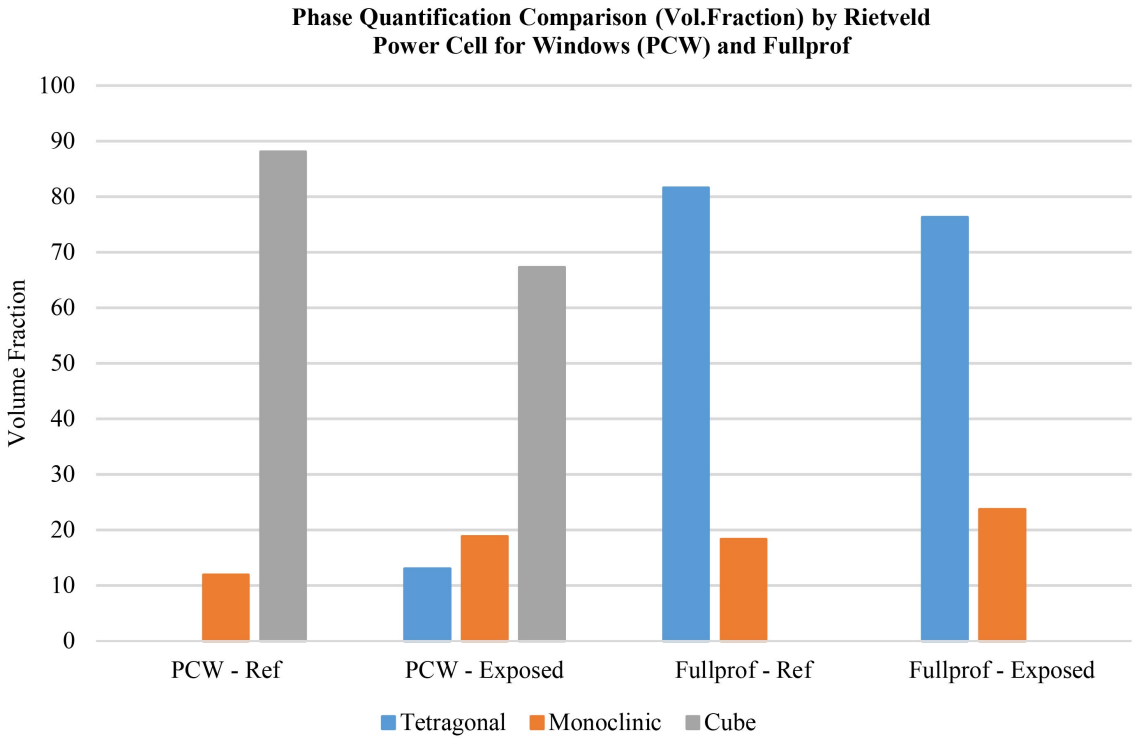


Figure 3. Histograms of the cubic, monoclinic, and tetragonal phase fractions before and after exposure to alternating relative humidity levels of 40% and 70% on a weekly basis for 85 weeks at 60˚C. The phase fractions were determined by Rietveld refinement using the PCW and FullProf programs. The histograms illustrate the increase in the monoclinic phase fraction following exposure.

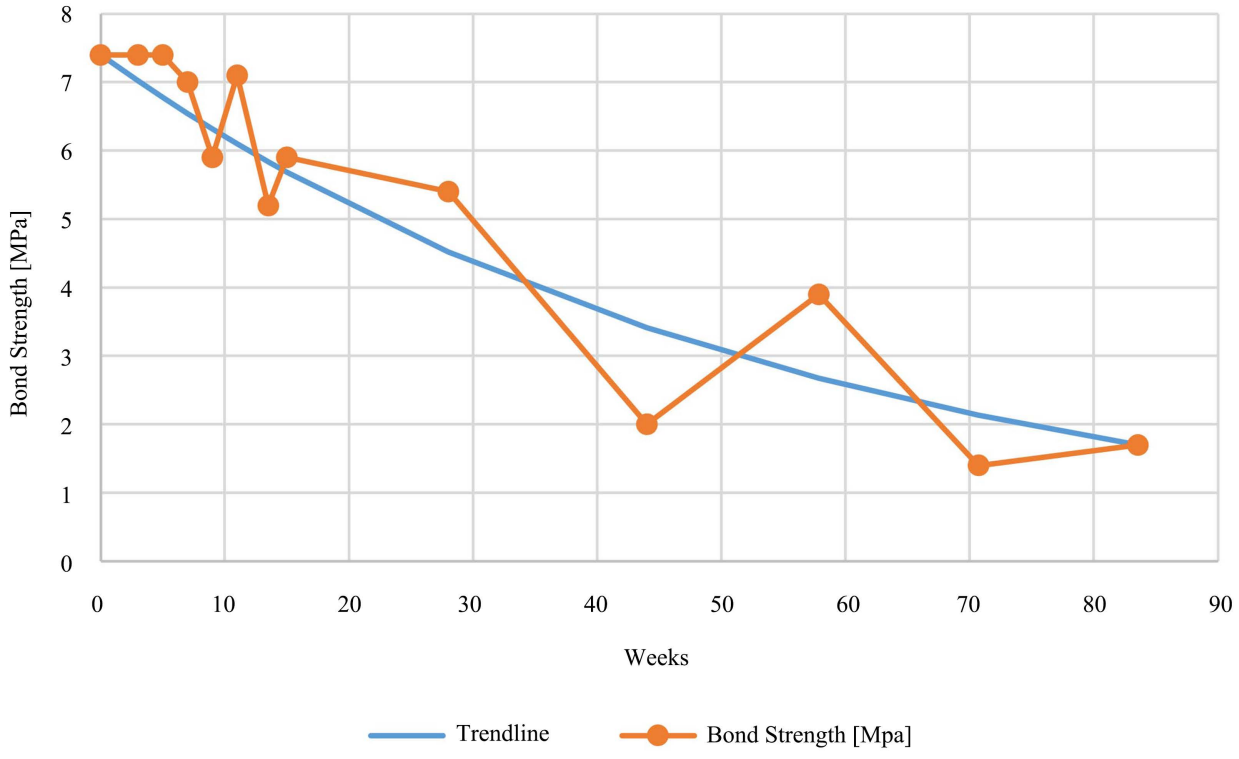


Figure 4. Time dependence of the bond strength of the samples in MPa.

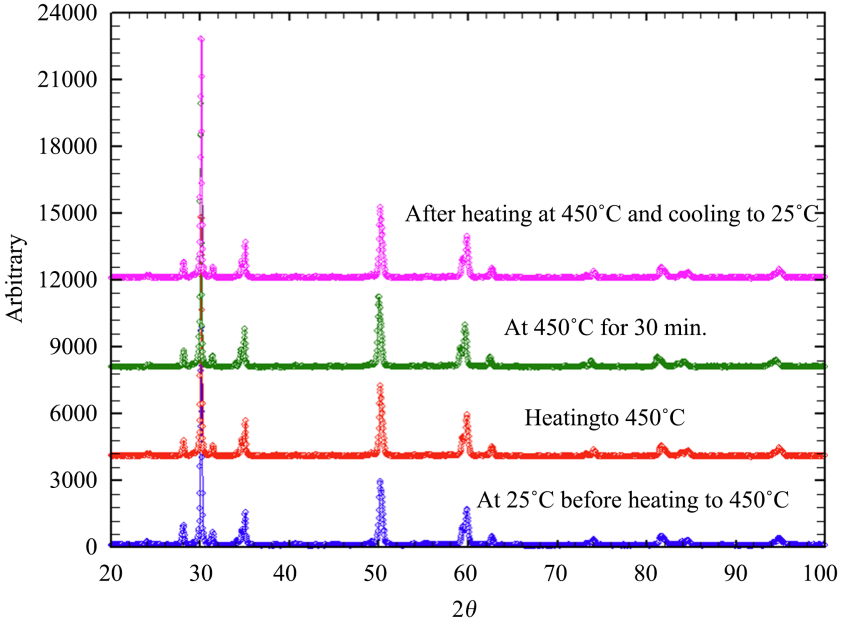


Figure 5. Stability verification of the phases identified by XRD in the sample subjected to alternating weekly exposure to 40% and 70% relative humidity over a total duration of 85 weeks. The sample was heated and degassed, and XRD patterns were collected at four stages: at 25˚C, after heating to 450˚C, after holding at 450˚C for 30 min, and after cooling back to 25˚C. The corresponding stages are indicated on the patterns. Rietveld refinement revealed no changes in phase composition.

4. Discussion

The stabilization mechanism of Metco 2460NS is based on the incorporation of yttrium (Y^{3+}) into the zirconia (ZrO_2) lattice, which creates oxygen vacancies and stabilizes the high-temperature zirconia phases at room temperature [13]. Pure zirconia exhibits three crystallographic polymorphs: monoclinic (m-ZrO_2), tetragonal (t-ZrO_2), and cubic (c-ZrO_2). According to the zirconia phase diagram shown in **Figure 6**, the monoclinic phase is stable at low temperatures, while the tetragonal and cubic phases are stable only at elevated temperatures [2]. The addition of yttria stabilizes the tetragonal and cubic phases by suppressing the phase transformations that would otherwise occur during cooling to room temperature. According to the PCW, in **Figure 6**, the red line corresponding to 18.8% monoclinic phase and the blue line corresponding to 11.9% monoclinic phase within the monoclinic + cubic phase zone are superimposed on the phase diagram to illustrate the change in phase relationships after exposure to moisture at 60˚C for 85 weeks. It should be noted that all specimens were initially heat treated according to the specified procedure, consisting of heating from room temperature to 450˚C at a rate of 5˚C - 8˚C/min followed by a hold at 450˚C [9]. Phase transformations in zirconia are often associated with significant volume changes. In particular, the increase of the monoclinic transformation is accompanied by a volume expansion of approximately 3% - 5%, which can generate internal stresses and promote microcracking. Exposure to moisture at 60˚C altered the phase balance

of the coating, primarily through the formation of a measurable tetragonal phase that was not observed after the initial coating preparation by PWC. As shown in Figure 2, the simultaneous increase monoclinic phase fractions correlate with the degradation of the bond strength presented in Figure 4. The stabilization of zirconia by yttria occurs through a defect chemistry mechanism at the atomic scale. Understanding this mechanism is essential for interpreting phase stability, Rietveld refinement results, and ionic conductivity in YSZ. Pure zirconia consists of Zr^{4+} cations and O^{2-} anions arranged to form electrically neutral ZrO_2. Yttria (Y_2O_3) introduces Y^{3+} ions into the zirconia lattice. Because the ionic radii of Y^{3+} (≈1.02 Å) and Zr^{4+} (≈0.84 Å) are relatively similar in eightfold coordination, Y^{3+} ions can substitute for Zr^{4+} lattice sites.

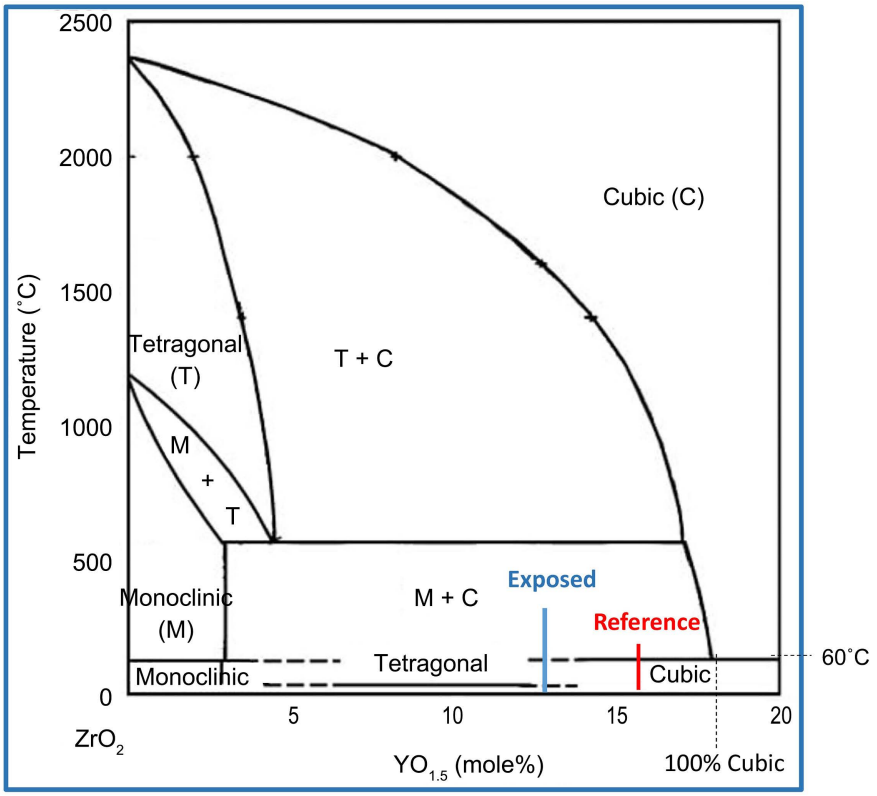


Figure 6. Phase Diagram of yttria-stabilized ZrO_2. Red line at 11.9% and (red) and 18.8% monoclinic phase calculated only in the M + C zone of the diagram and relative to this zone, after exposed for 85 weeks to moisture at 60˚C (blue). Diagram from Witz *et al.* [2].

However, each substitution introduces a charge deficiency of one positive charge. To maintain charge neutrality, the lattice compensates by creating oxygen vacancies according to:

$$2Y^{3+} + V_O \leftrightarrow 2Zr^{4+}$$

The formation of oxygen vacancies leads to local lattice relaxation, with neighboring oxygen ions and cations slightly shifting their positions. These local distortions reduce lattice strain and lower the free energy of the tetragonal and cubic

structures, thereby stabilizing these high-temperature phases at room temperature. As illustrated in Figure 6, yttria-stabilized tetragonal and cubic zirconia can therefore persist under ambient conditions. Prolonged exposure to moisture, however, modifies this equilibrium. Water molecules interact with the oxygen-vacancy network and may promote local redistribution or depletion of yttrium from vacancy-rich regions. As a result, the stabilizing effect of the yttria-vacancy defect pairs is reduced. The present study indicates that humidity exposure at 60˚C promotes both the formation of the tetragonal (pseudo-cubic) phase and an increase in the monoclinic phase fraction. When yttrium diffuses away from vacancy-rich regions, local phase stabilization decreases and the tetragonal phase becomes thermodynamically unstable. Consequently, tetragonal zirconia may transform to the monoclinic phase, a transformation accompanied by a volume expansion of approximately 3% - 5%. The resulting local stresses can promote microcrack initiation and propagation. This mechanism is widely recognized as a key contributor to hydrothermal aging and low-temperature degradation of YSZ [13] [14]. The associated transformation-toughening behavior relies on stress-induced phase transformation and directly influences the bond strength reported in this work following prolonged exposure to a high-moisture environment at 60˚C.

5. Conclusions

To conclude this study, the following findings can be highlighted:

1) Long-term exposure of yttria-stabilized zirconia (YSZ)-based Metco 2460NS abradable coatings to alternating weekly relative humidity levels of 40% and 70% at 60˚C over 85 weeks results in a measurable reduction in bond strength.

2) Phase quantification of the cubic, tetragonal, and monoclinic zirconia phases before and after 85 weeks of exposure revealed an increase in the monoclinic phase fraction, the formation of a tetragonal phase, and a corresponding decrease in the cubic phase content.

3) Exposure to alternated weekly between 40% and 70%, for 85 weeks moisture at 60˚C promotes changes in the oxygen-vacancy distribution and may induce local yttrium redistribution, thereby reducing the stabilization of the zirconia lattice.

4) The observed phase evolution is attributed to the yttria-stabilization mechanism, which involves the formation of oxygen vacancies that compensate for the charge imbalance created when Y^{3+} ions substitute for Zr^{4+} ions in the zirconia lattice.

5) As a result, the cubic phase becomes less stable, leading to an increase in the monoclinic phase fraction. The associated phase transformation induces a volume expansion of approximately 3% - 5%, directly contributing to the degradation of the coating's bond strength.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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