



Characterization of ZrO_2 - Y_2O_3 Thermal Barrier Coatings By Raman Spectroscopy

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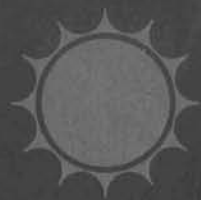
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CHARACTERIZATION OF ZrO_2 - Y_2O_3 THERMAL BARRIER COATINGS
BY RAMAN SPECTROSCOPY

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ABSTRACT

Raman spectroscopy is shown to be a useful tool for the observation of phase transitions in Y_2O_3 stabilized ZrO_2 thermal barrier coatings. Data have been collected in situ at temperatures up to 1050°C as a function of Y_2O_3 content. Large hysteresis effects are observed in phase transition temperatures.

INTRODUCTION

Significantly improved gas turbine operating efficiencies can be achieved by coating turbine blades with a ceramic thermal barrier.¹ This coating acts to reduce metal surface temperatures and provide a barrier to the ingress of corrosive species in the combustion products. As a result, higher inlet temperatures can be tolerated, leading to improved efficiencies with the use of existing high temperature, high strength alloys.

Numerous ceramics are being considered for use as thermal barriers. Zirconia-based coatings containing 8-12 wt% Y_2O_3 have received the most extensive investigation. These tetragonal/cubic phase Y_2O_3 stabilized coatings have been utilized to avoid potentially severe cracking associated with the large volume change ($\sim 6\%$) transition to the monoclinic phase inherent in pure ZrO_2 . Nevertheless, failure of the ZrO_2 based coatings is usually attributed to destabilization and the associated phase transitions initiated by either thermal cycling or reaction with fuel impurities.²

Three phases have been reported for ZrO_2 based ceramics containing less than 20 wt% Y_2O_3 .^{3,4} The exact compositions and temperature ranges over which these phases exist is not well known, as shown by the numerous lines in Fig. 1 representing the tentative phase boundaries. At low temperatures ($< 1100^\circ C$) and low Y_2O_3 contents (< 5 wt Y_2O_3), a monoclinic structure is reported to be the predominant stable phase. Upon increasing the temperature or the Y_2O_3 content, a tetragonal structure predominates. Further increases in the Y_2O_3 content lead to a cubic structure. The phase transition

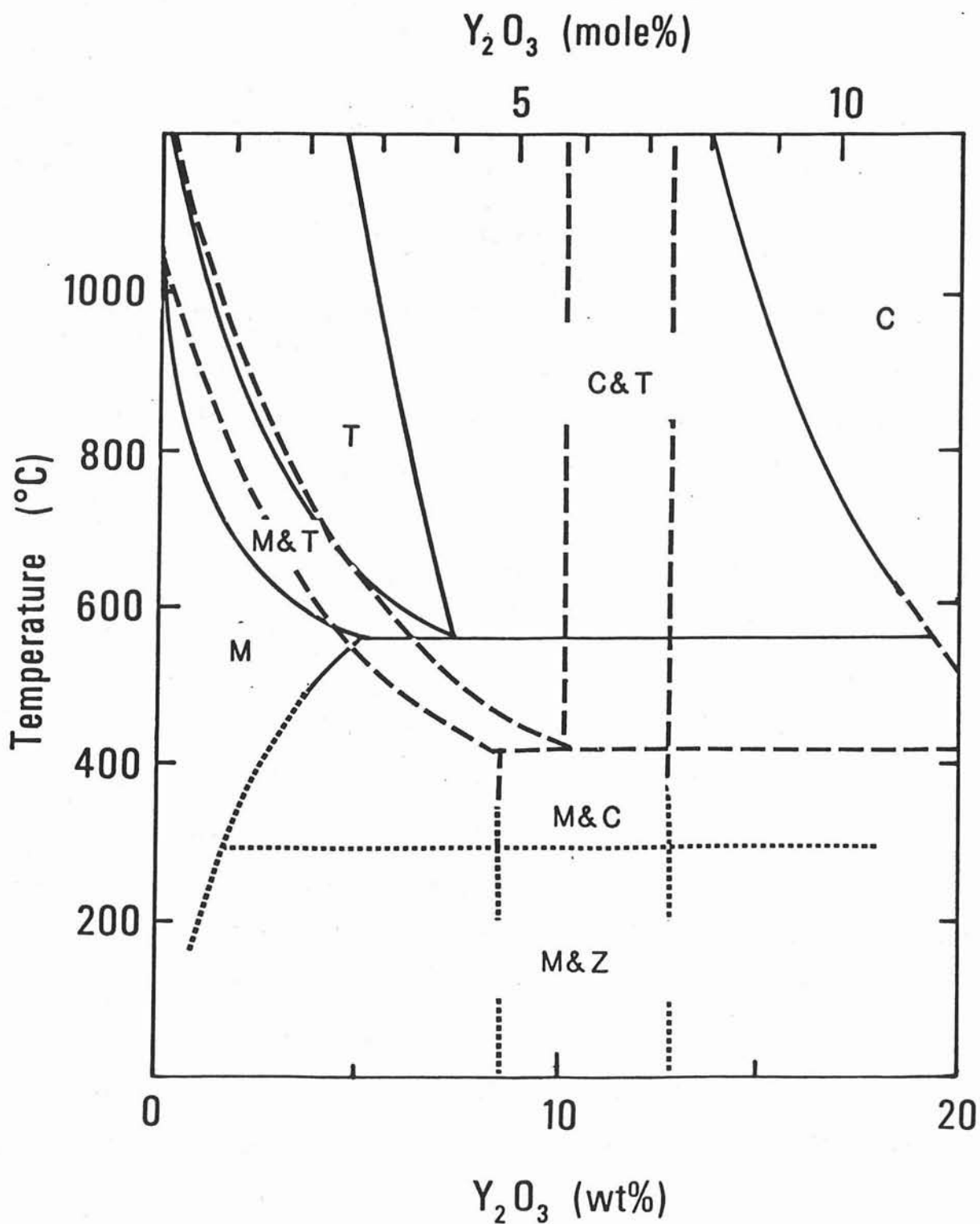


Figure 1. The published phase diagram for ZrO₂-Y₂O₃. The solid lines are from Ref. 3 and the dashed lines are from Ref. 4. The dotted lines represent phase field extrapolations. Phases are identified as cubic, C; Tetragonal, T; Monoclinic, M; and Zr₃Y₄O₁₂, Z.

from either the cubic or tetragonal phases to the monoclinic structure results in a large volume change and potentially severe cracking.

In order to improve the performance of these coatings, a more precise knowledge of the phase diagram is required to elucidate the processes occurring during thermal cycling and the modes of the subsequent failure of the ZrO_2 based coatings. Furthermore, techniques are required to determine the uniformity of Y_2O_3 content in commercially prepared coatings. This paper discusses the feasibility of utilizing spontaneous Raman spectroscopy to determine the $ZrO_2 - Y_2O_3$ phase diagram as well as to monitor in-service phase transitions of ZrO_2 based thermal barrier coatings.

EXPERIMENTAL

Ceramic ZrO_2 disks containing nominally 1.0, 1.5, 2.5, 4.0, 6.0, 8.0, 13.8, and 16.0 wt% Y_2O_3 were obtained from Zircar Products, Inc. Powdered starting materials were prepared by co-precipitation within a matrix and calcining at 1200°C. The resultant $<0.01 \mu m$ powder was cold pressed at 12,000 psi and fired in air. Fired disks were furnace cooled from sintering temperature. Typical impurity levels are given in Table I. Thermal barrier coatings were physical vapor deposited on an In 738 substrate with a NiCrAlY bond coating.⁵

A conventional, right-angle, quasi-backscattering configuration employing either a cw argon or krypton laser, a triple grating monochromator, and GaAs photomultiplier with photon counting electronics was utilized to obtain room temperature Raman spectra of the $ZrO_2 - Y_2O_3$ disks as a function of Y_2O_3 content. For measurements as a function of temperature, a similar scattering geometry was employed within a laboratory furnace in conjunction with an

TABLE I

TYPICAL IMPURITY LEVELS (WT %)

ZrO ₂	84.4	CaO	0.1
Y ₂ O ₃	11.7	MgO	0.1
HfO ₂	1.6	Fe ₂ O ₃	0.01
Al ₂ O ₃	0.2	H ₂ O	1.7
Cl	0.3		

optical multi-channel Raman analyzer consisting of a three-stage grating spectrograph and SIT vidicon detector. Using the multichannel approach, spectra could be recorded in times as short as 15 ms. Thus, spectral or sample symmetry changes occurring during the acquisition of individual Raman spectra were virtually eliminated. The sample area probed was rectangular with dimensions of $\sim 50 \mu\text{m} \times 2.5 \text{ mm}$.

Initial Raman data obtained from the fired $\text{ZrO}_2\text{-Y}_2\text{O}_3$ ceramics as a function of Y_2O_3 content at room temperature clearly indicate the distinct nature of the Raman spectra obtained from the monoclinic, tetragonal, and cubic phases, as shown in Fig. 2a, 2b, and 2c, respectively. The data are in good agreement with a previous Raman analysis⁶ of pure ZrO_2 for the monoclinic, tetragonal, and cubic phases, respectively. Nevertheless, the symmetry designations in Fig. 2 are based on independent room temperature x-ray diffraction results.

Spectra obtained at room temperature from the Zircar disks having varying Y_2O_3 contents within the monoclinic, tetragonal, and cubic symmetry groups were essentially identical. The ceramic disks containing 1.0, 1.5, and 2.5 wt% Y_2O_3 had the monoclinic structure at room temperature. The predominant phase in the 4.0, 6.0, and 8.0 wt% Y_2O_3 samples was tetragonal. Both the 13.8 and 16.0 wt% disks were found to have the cubic structure.

Figure 3 shows the Raman data from $\text{ZrO}_2\text{-1 wt% Y}_2\text{O}_3$ obtained at 25°C and 1050°C using the single channel Raman apparatus. Upon heating from room temperature to 1050°C, the sharp peaks associated with the monoclinic phase become less clearly resolved because of thermal broadening. Nevertheless, the frequency shifts of the characteristic vibrational modes are only slightly modified, indicating that no phase transformation has occurred within this temperature range. As can be seen in Fig. 1, the published phase diagrams

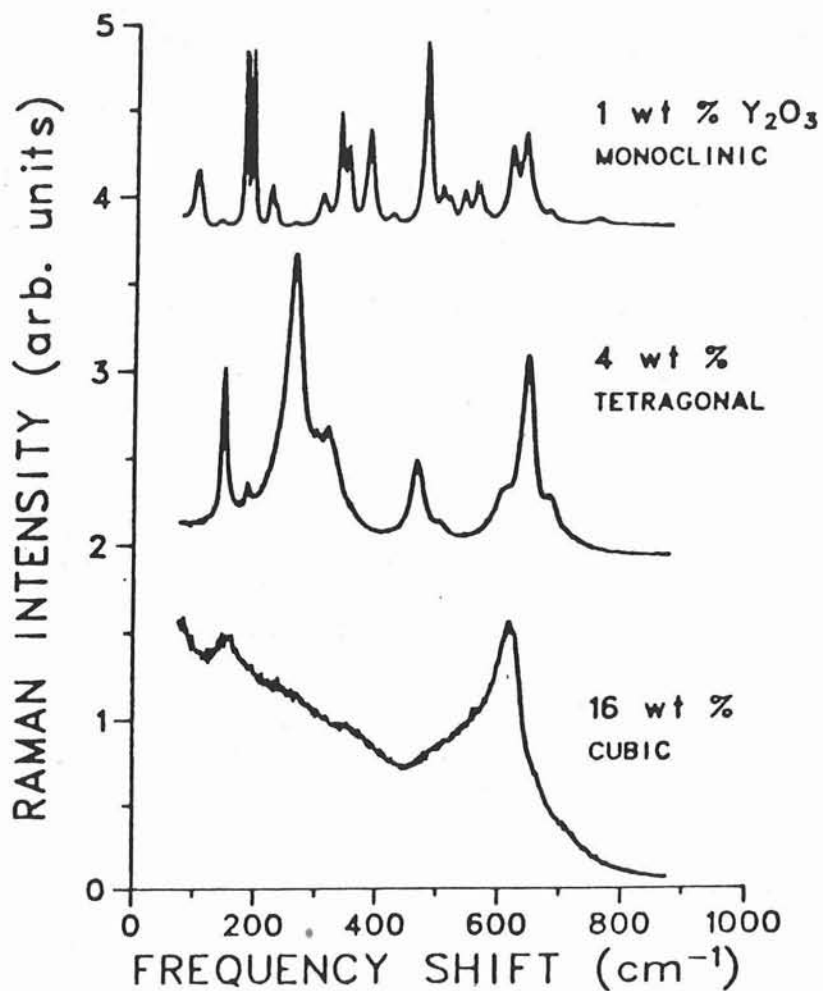


Figure 2. Raman data obtained at room temperature from $\text{ZrO}_2 - \text{Y}_2\text{O}_3$ showing the characteristic spectra of the monoclinic, tetragonal, and cubic phases. Symmetry designations were confirmed by x-ray diffraction analysis.

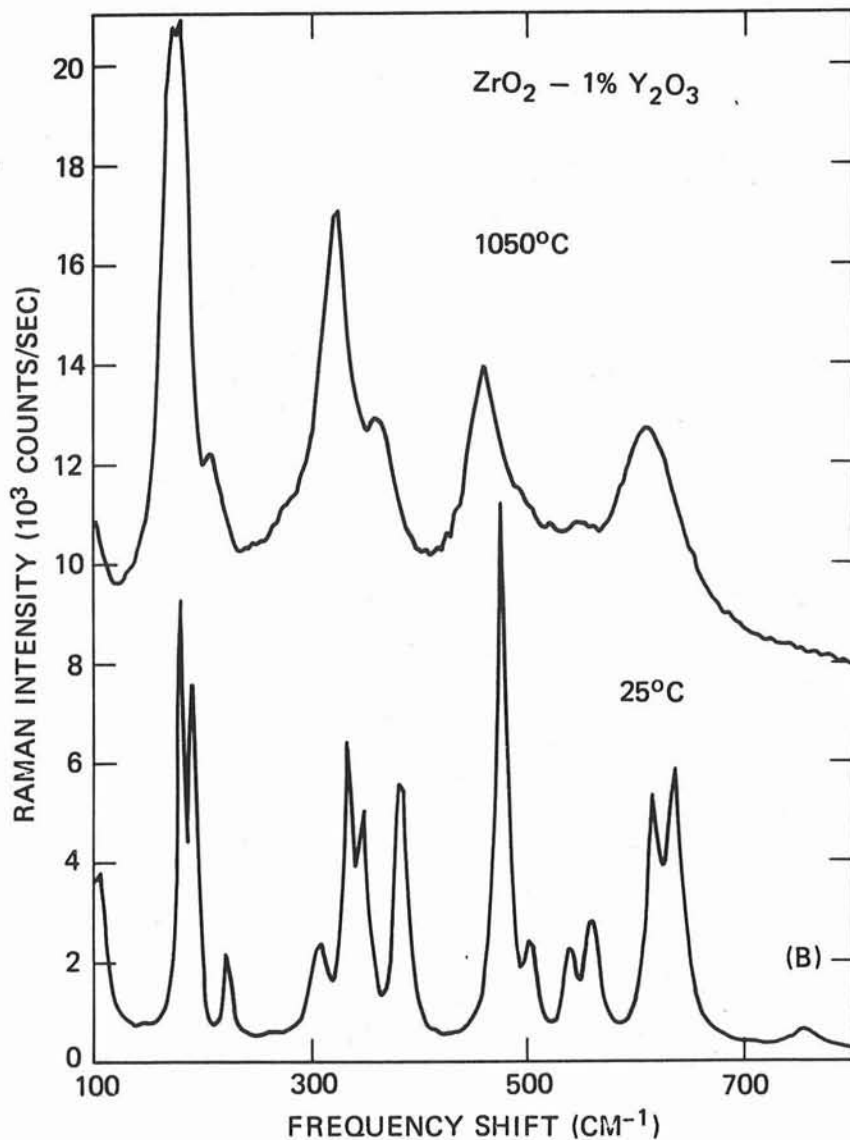


Figure 3. Raman spectra from monoclinic $ZrO_2 - 1\text{ wt}\% Y_2O_3$ at $1050^\circ C$ and $25^\circ C$ showing broadening of the Raman peaks at high temperature.

would have predicted a phase transition. In contrast, a transition from the monoclinic to tetragonal phase was clearly observed to begin at approximately 790°C during heating of the ZrO_2 -2.5 wt% Y_2O_3 sample at 6°/min, as shown in the multichannel Raman data of Fig. 4(a). The appearance of a strong mode at 275 cm^{-1} provides the most dramatic indication of tetragonal symmetry. This observed start of the phase transition is above the 630°C previously reported.^{1,2} However, it is expected that some hysteresis will exist in a ceramic system at these temperatures, because of the slow rate of the solid state transformation. In fact, large hysteresis effects were observed using the Raman technique as the ZrO_2 -2.5 wt% Y_2O_3 sample was cooled from 1000°C at approximately 6°/min. Figure 4(b) indicates that the tetragonal to monoclinic transition occurs at approximately 600°C upon cooling, compared to the 790°C value found upon heating. Hysteresis effects have also been observed by Raman spectroscopy in the ZrO_2 - HfO_2 system.⁷

Analysis of the ZrO_2 based thermal coatings prepared by Airco-Temescal indicated that these coatings were either tetragonal or cubic. Coatings containing 8 wt% Y_2O_3 applied by physical vapor deposition were tetragonal, as can be seen in Fig. 5. The Raman data for the 12 wt% Y_2O_3 coating suggests a cubic symmetry with minor indications of the tetragonal phase. Complementary x-ray diffraction analysis showed that these coatings exhibit strong preferred orientations, the specific crystallographic orientation varying from sample to sample. In this technique, where deposition is achieved by vaporization of an oxide melt by an electron beam, the orientation of the initial oxide deposit apparently strongly influences the subsequent deposition process.

Another characteristic of the physical vapor deposited thermal barrier coatings was the existence of strong fluorescence bands at ~523, 543, and 562 nm which were absent from the purer ZrO_2 - Y_2O_3 disks. The occurrence

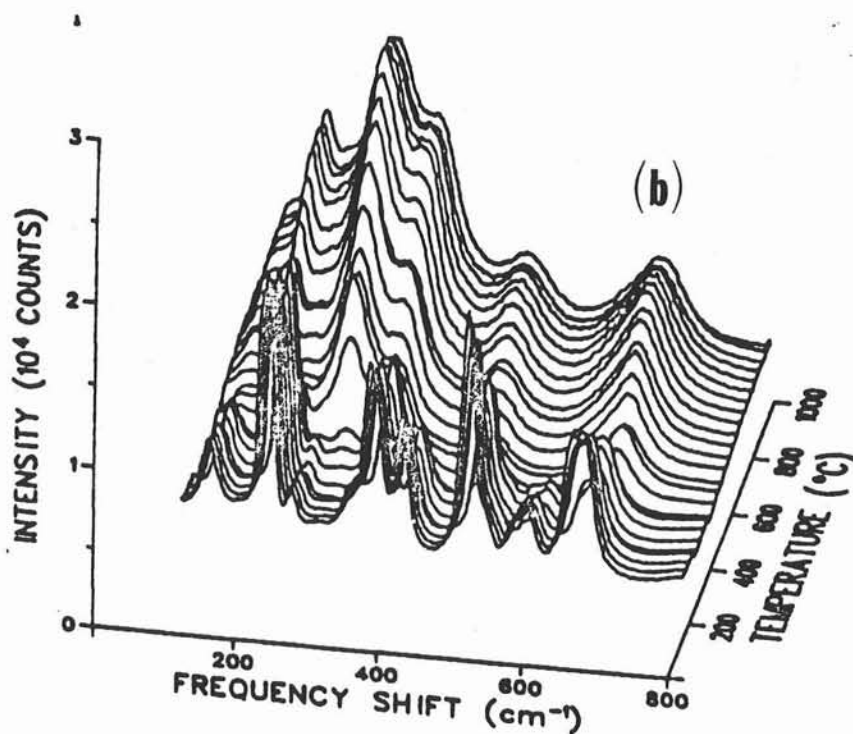
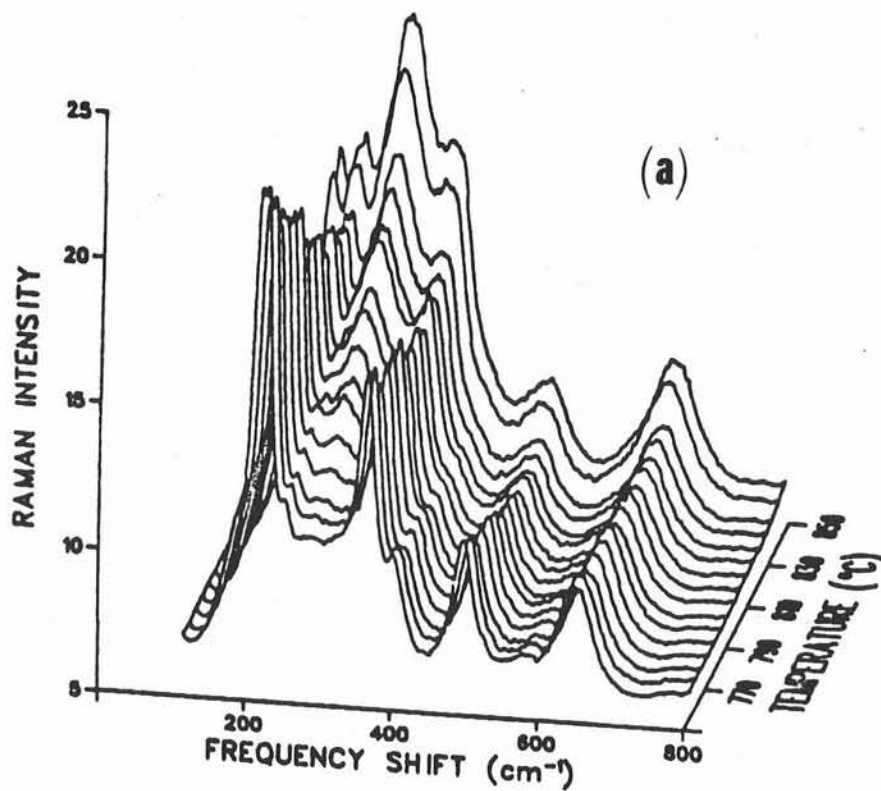


Figure 4. Multichannel Raman data obtained from $ZrO_2-2.5 \text{ wt\% } Y_2O_3$ as a function of temperature showing the occurrence of the monoclinic to tetragonal phase transition during heating (a) and cooling (b) at a rate of $6^\circ\text{C}/\text{min}$. The mode at $\sim 275 \text{ cm}^{-1}$ is the most dramatic indication of tetragonal symmetry.

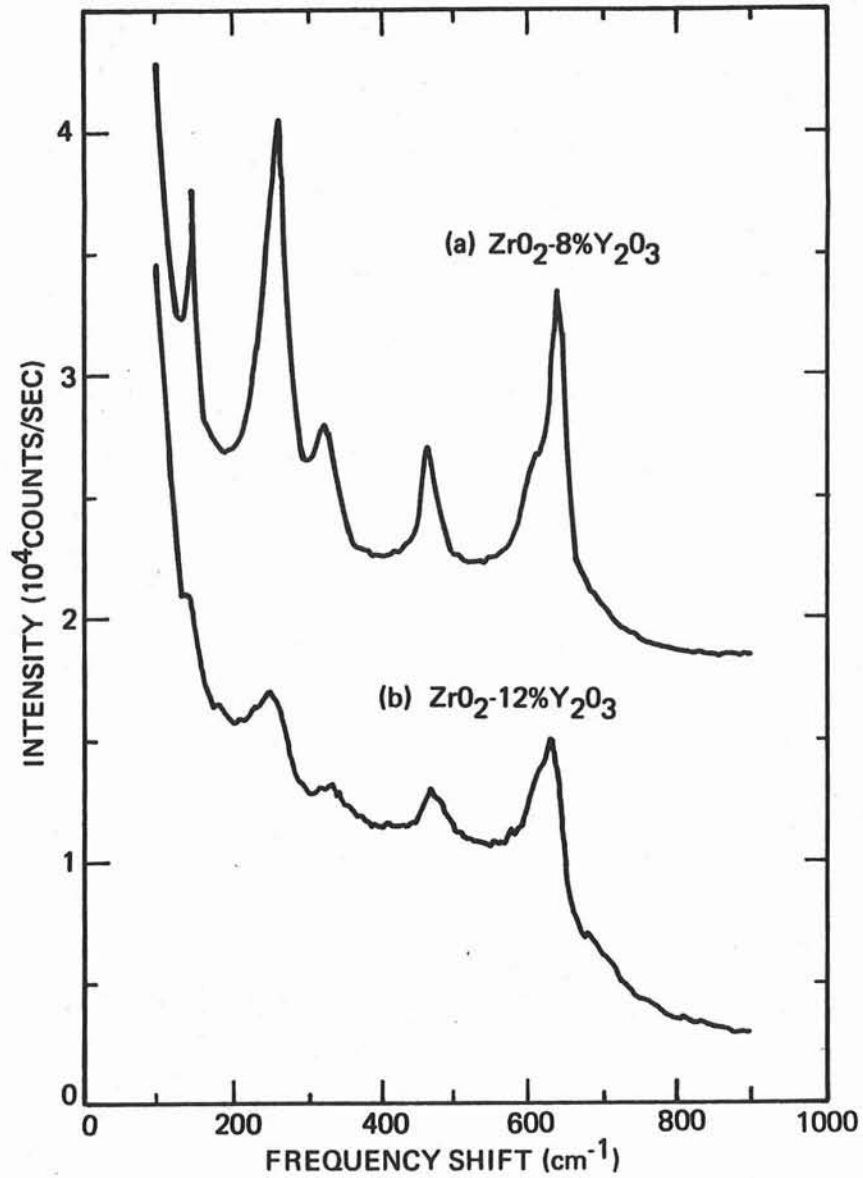


Figure 5. A comparison of room temperature Raman spectra from ZrO₂ based thermal barrier coatings containing 8% and 12% Y₂O₃. The 8% coating is clearly tetragonal, while the 12% material is cubic with a minor quantity of tetragonal phase.

of these bands is a possible indication of rare earth impurities in the coatings and the source material. If detrimental effects due to ceramic impurities are identified during further investigations of $ZrO_2-Y_2O_3$, the observed fluorescence could be used effectively to determine the impurity species and to screen the source materials.

Extension of these preliminary Raman results to in-service mapping of these phase homogeneity of ZrO_2 based coatings should be possible. For example, utilizing the two dimensional capability of the vidicon array, as many as 500 spectra could be obtained nearly simultaneously as a function of position along the $\sim 50 \mu m \times 2.5 mm$ laser-illuminated focal area. Thus, high resolution mapping of the phases present along the illuminated line could be achieved.

CONCLUSION

Raman spectroscopy has been found to be a sensitive technique for determining the phase symmetry of $ZrO_2-Y_2O_3$ thermal barrier coatings. Unique Raman spectra are obtained for the three structural phases present in the system. While somewhat limited by thermal broadening, the technique can be utilized in situ at high temperatures. In agreement with previous results, large hysteresis effects have been observed in the temperatures at which phase transitions occur, depending upon whether the sample was being heated or cooled. Future studies will be aimed at a more accurate determination of the $ZrO_2-Y_2O_3$ phase diagram than is currently available, and at characterizing thermal barrier coatings as a function of deposition conditions and temperature cycling.

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