

# Phase transformations in air plasma-sprayed yttria-stabilized zirconia thermal barrier coatings

## Transformaciones de fase en recubrimientos de barrera térmica de zirconia estabilizada con yttria depositados mediante aspersión por plasma atmosférico

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### Abstract

Phase transformations in air plasma-sprayed thermal barrier coatings composed of  $ZrO_2 - 8 \text{ wt.}\% Y_2O_3$  (zirconia - 8 wt.% yttria) are studied using X-Ray diffraction and Rietveld refinement measurements. Samples of TBC deposited onto Inconel 625 substrate were fabricated and heat treated at two different conditions: exposition to  $1100^\circ\text{C}$  up to 1000 hours and exposition to temperatures between  $700^\circ\text{C}$  and  $1100^\circ\text{C}$  during 50 hours. According to Rietveld refinement measurements, the content of the cubic phase in the top coat increases with time and temperature; it starts at 7.3 wt.% and reaches 15.7 wt.% after 1000 hours at  $1100^\circ\text{C}$ . The presence of a cubic phase in high amounts is undesirable due its lower mechanical properties compared with the tetragonal phase. After 800 hours of exposure to high temperature, the amount of  $Y_2O_3$  in the tetragonal phase reduces to 6.6 wt.% and a fraction of this phase transforms to a monoclinic structure during cooling. The monoclinic phase reached 18.0 wt.% after 1000 hours. This phase is also undesirable, not only due to its higher thermal conductivity, but also because the tetragonal-to-monoclinic transformation implies a volume change of circa 5%, which favors crack formation and propagation and compromises the coating integrity.

**Keywords:** Thermal Barrier Coating (TBC); Heat Treatment; Phase Transformation; Rietveld Analysis.

### Resumen

En este trabajo, las transformaciones de fase en Recubrimientos de Barrera Térmica (TBC) constituidos por  $ZrO_2 - 8 \text{ wt.}\% Y_2O_3$  (zirconia - 8 wt.% yttria) fueron estudiados a través de Difracción de Rayos X (XRD) y refinamiento Rietveld. Las muestras de TBC fueron depositadas mediante aspersión por plasma atmosférico sobre un sustrato tipo Inconel 625 y fueron tratadas térmicamente con dos condiciones diferentes: en la primera se utilizó una temperatura de  $1100^\circ\text{C}$  con tiempos de exposición entre 1 hora y 1000 horas; en la segunda las muestras fueron sometidas a temperaturas entre  $700^\circ\text{C}$  y  $1100^\circ\text{C}$  durante 50 horas. De acuerdo a los resultados obtenidos mediante refinamiento Rietveld el contenido de fase cúbica en el recubrimiento (TC) se incrementa con el tiempo y la temperatura, desde 7.3 wt.% hasta 15.7 wt.% después de 1000 horas a  $1100^\circ\text{C}$ . La fase cúbica en grandes cantidades es indeseable debido a que presenta inferiores propiedades mecánicas cuando se compara con la fase tetragonal. Después de 800 horas de exposición a alta temperatura, el contenido de  $Y_2O_3$  en la fase tetragonal se reduce hasta 6.6 wt.% y una fracción de la fase tetragonal transforma a monoclinica durante el enfriamiento. La fase monoclinica alcanza 18.0 wt.% después de 1000 horas. Esta fase es también indeseable porque además de tener una mayor conductividad térmica, la transformación de tetragonal a monoclinica viene acompañada de un cambio volumétrico de alrededor de 5% que promueve la formación y propagación de grietas, las cuales comprometen la integridad del recubrimiento.

**Palabras clave:** Recubrimiento de Barrera Térmica (TBC); Tratamiento Térmico; Transformaciones de fase; Refinamiento Rietveld.

### 1. Introduction

Thermal barrier coatings (TBCs) are multilayered systems widely used in gas turbines to increase efficiency and durability [1-4]. These coatings consist of three layers deposited onto a Base Substrate (BS): the Top Coat (TC), the Bond Coat (BC) and the Thermally Grown Oxide (TGO)

layer (see Fig. 1). Base substrates are usually Ni-based superalloys that offer good mechanical strength and excellent corrosion, oxidation and erosion resistances at high temperatures [5-7]. They contain significant amounts of alloying elements such as Cr, Mo, Al, Ti, Fe and C, which favor intermetallic compounds precipitation [8,9]. Two types of BCs are commonly used: the Platinum- modified Nickel

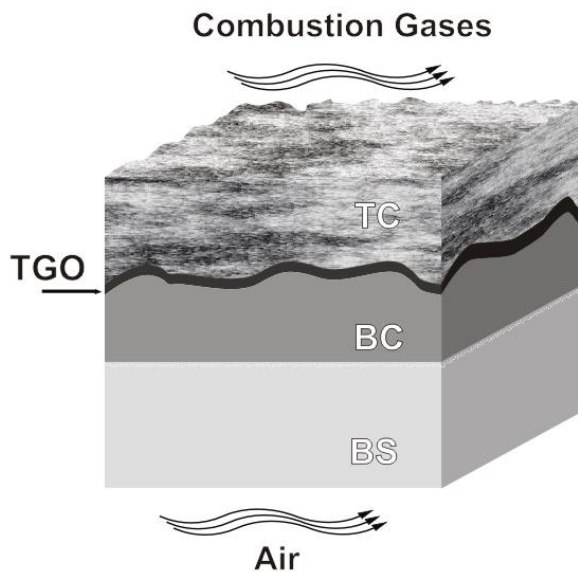


Figure 1. Schematic diagram of a Thermal Barrier Coating applied by Air Plasma Spray (APS). The TC faces combustion gases. The BS is air-cooled to increase the temperature gradient and, therefore, the efficiency.

Aluminide (PtNiAl) and MCrAlY alloys, where M refers to one or more of the elements Co, Ni and Fe. The BC is a metallic layer that initially provides adherence between the TC and the substrate [10,11]. During operation at high temperatures, aluminum diffuses from the BC and reacts to form a barrier layer, known as the TGO. Once the TGO is formed, the BC serves as the anchoring layer between the TC and the TGO. The TGO provides the barrier to oxygen diffusion to avoid substrate oxidation. However, many of the failure mechanisms in TBCs are related to the TGO formation and growth [12-16].

The TC is usually composed by Yttria ( $Y_2O_3$ ) stabilized Zirconia ( $ZrO_2$ ), and serves as the main defense mechanism of gas turbines against erosion and corrosion. The TC has low thermal conductivity that reduces the temperature of the bond coat up to  $500^\circ C$  with a thickness of some hundred microns. It must be stabilized in order to maintain its tetragonal structure at room temperature and also to keep thermal properties constant (conductivity and thermal expansion coefficient) in the range of working temperatures. To accomplish this stabilization, some elements such as Hafnium (Hf) and Yttrium (Y), are commonly added [17-20]. The Yttrium ( $Y^{+3}$ ) and Hf ( $Hf^{+4}$ ) ions replace the zirconium ( $Zr^{+4}$ ) ions in the lattice cell inducing changes in the crystal structure. These changes stabilize the tetragonal phase and decrease the thermal conductivity. In the first case, the  $Y^{+3}$  ions produce oxygen vacancies in the lattice [18], while the  $Hf^{+4}$  ions, which are chemically similar and have comparable ionic radius to  $Zr^{+4}$  ions, are almost twice as massive and generate a lattice disorder. In both cases, thermal conductivity is reduced due to an un-harmonic scatter of the charge carriers in the ceramics at high temperature, i.e. un-harmonic phonon scatter phenomenon [19,21].

Zirconia without stabilizers can exist in three different phases [18,22]: cubic, tetragonal and monoclinic, see the phase diagram [23] in Fig. 2. The tetragonal-to- monoclinic

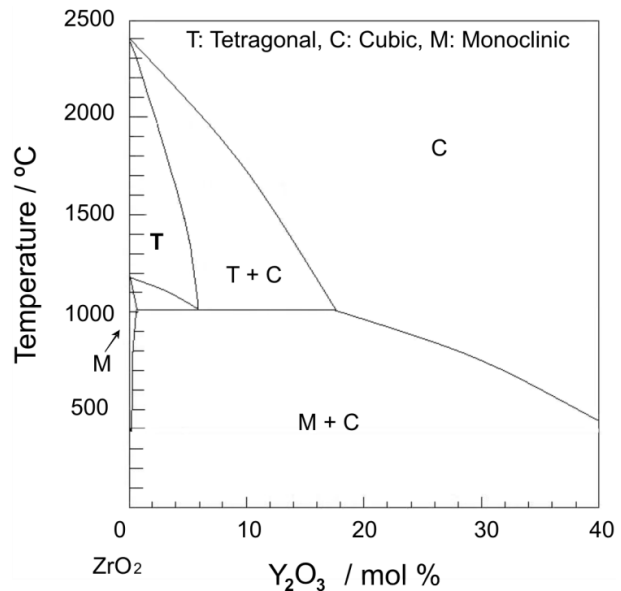


Figure 2. Portion of the phase diagram of  $ZrO_2 - Y_2O_3$  system [21].

transformation is undesirable because it is accompanied by a volumetric change of circa 5%, which causes a detrimental effect to the TBC due to crack nucleation and propagation [24,25]. In addition, the tetragonal phase presents excellent mechanical and thermal properties compared with those of the monoclinic phase [2,19,26,27]. The tetragonal-to-monoclinic transformation is avoided with the addition of 6 to 8 wt.% of Yttria [18].

Two different processes are currently used to deposit Yttria stabilized Zirconia: Electron Beam-Physical Vapor Deposition (EB-PVD) and Air Plasma Spray (APS). In both cases, the rapid cooling results in a metastable tetragonal phase ( $t'$ ) rather than a stable tetragonal [28,29]. According to some studies, during exposure to high temperature and during cycling operations, Yttrium (Y) diffuses from the  $t'$  phase to stabilize the cubic phase [30,31]. Consequently, a monoclinic phase appears from the Y-depleted tetragonal phase during cooling.

The presence of both, cubic phase in high amounts and monoclinic phase is undesirable due to their lower mechanical properties compared with the tetragonal phase. Also, the volumetric changes associated with the phase transformations favor crack generation and propagation which compromise the coating integrity. Therefore, understanding these transformations is essential to find alternatives to improve the TBC's lifetime.

In this work, phase transformations in an APS-deposited TC under two different sets of heat treatments are studied using X-Ray Diffraction (XRD) and semi-quantitative Rietveld refinement. The paper is organized as follows: in Section 2, the experimental procedure, methods and materials are presented. Section 3 describes a summary of the results, where the effects of exposure time at  $1100^\circ C$  are analyzed first and then the phase dynamics for different exposure temperatures during 50 hours is discussed. The most important conclusions are summarized at the end.

## 2. Experimental procedure

The TBC samples are composed of a  $ZrO_2 - 8 \text{ wt.}\%$   $Y_2O_3$  TC applied by APS onto a NiCrCoAlY BC, both layers having thicknesses around  $300 \mu\text{m}$ . The BC layer was deposited onto a nickel-base substrate, namely Inconel 625. The dimensions of the TBC samples were  $10 \text{ cm} \times 10 \text{ cm}$ , extracted from plates of  $30 \text{ cm} \times 30 \text{ cm}$ . The samples were cut with a precision saw operating at 4000 RPM. Thereafter, some samples were heated at a rate of  $18 \text{ }^\circ\text{C}/\text{min}$  and maintained at  $1100^\circ\text{C}$  between 1 and 1000 hours, while other samples were thermally treated for 50 hours at  $700^\circ\text{C}$ ,  $800^\circ\text{C}$ ,  $900^\circ\text{C}$  and  $1000^\circ\text{C}$ ; In all cases, the samples were cooled in air.

Sample preparation included grinding with No. 400 and No. 600 emery papers for 5 minutes, followed by polishing by cloths with abrasive diamond suspensions containing particles with  $12 \mu\text{m}$ ,  $6 \mu\text{m}$ ,  $3 \mu\text{m}$  and  $1 \mu\text{m}$  in average size. The polishing time for the first three suspensions was 15 minutes, while 60 minutes were required for the  $1 \mu\text{m}$  suspension. A complete characterization of this material with similar heat treatment conditions has been reported in previous works [32,33].

The phase characterization was carried out in a Panalytical X Pert Pro MPD X-Ray Diffractometer with a  $\text{CuK}\alpha$  ( $\lambda = 1.5406 \text{ \AA}$ ) radiation gun within a  $20^\circ < 2\theta < 100^\circ$  range and step of  $0.013^\circ/\text{seg}$ . Rietveld semi-quantitative measurements were made to account the phase changes at different temperature conditions. To ensure reliability of the results, two replicas of each sample were also measured. The software used to perform the Rietveld refinements was X'Pert High Score Plus Version 2.2a by PANalytical B.V. It is well known that the tetragonal lattice parameters change depend on the  $Y_2O_3$  content. Therefore, Rietveld measurements were also performed to determine the lattice tetragonal parameters  $a$  and  $c$ , in order to determine the amount of  $Y_2O_3$  in this phase. The amount of  $Y_2O_3$  in the tetragonal phase, for each heat treatment condition, was determined using the following relation [22,34]:

$$YO_{1.5}(\text{mol.}\%) = \frac{1.0225 - (c/a)}{0.001311}, \quad (1)$$

where  $a$  and  $c$  are the lattice tetragonal parameters in nanometers. This expression was derived by H.G. Scott in 1975 [22], based on the change of lattice parameters of Yttria Stabilized Zirconia powders with different  $YO_{1.5}$  content; it was corrected empirically by Ilavsky and Stalick [34] to improve the fit throughout the annealing process to use it in a wide range of samples [35].

## 3. Results and analysis

In the as-sprayed condition, the TC was a mixture of  $92.7 \text{ wt.}\%$  tetragonal and  $7.3 \text{ wt.}\%$  cubic phases. In this condition, the  $Y_2O_3$  content in the tetragonal phase was  $7.53 \text{ wt.}\%$  ( $\sim 7.53 \text{ mol}\%$   $YO_{1.5}$ ) which is within the recommended range for which the tetragonal-to-monoclinic transformation

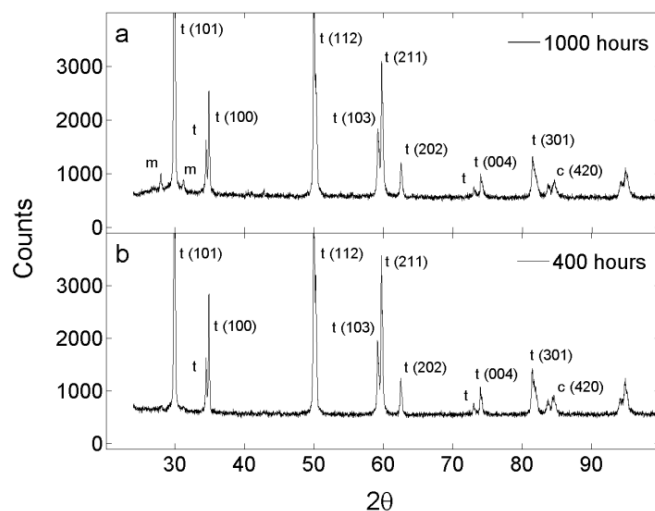


Figure 3. X-Ray diffractograms of TC treated at  $1100^\circ\text{C}$  for different exposure times. a) 1000 hours. b) 400 hours. Measurements performed at room temperature, after the heat treatments

is prevented after cooling [18]. Some researchers have reported a TC consisting exclusively of the tetragonal phase [31] and others have found considerable amounts of the monoclinic phase in the as-sprayed condition [34,36]. The differences in the initial compositions and microstructure depend, not only on the stabilizers' content, but also on the feedstock [34] and the presence of unmelted or partially melted particles [36].

### 3.1. Effect of exposure time at $1100^\circ\text{C}$

Fig. 3 presents two x-ray diffractograms for the samples thermally treated during 1000 hours and 400 hours at  $1100^\circ\text{C}$ , respectively. It can be observed that the monoclinic phase appears in the sample treated for 1000 hours. The evolution of the TC phases after the exposure to  $1100^\circ\text{C}$ , measured through Rietveld refinement, is shown in Fig. 4. The tetragonal phase decreases from  $97.3 \text{ wt.}\%$  to  $66.3 \text{ wt.}\%$  after 1000 hours, while the cubic phase increases from  $7.3 \text{ wt.}\%$  to  $15.7 \text{ wt.}\%$ . At 800 hours, the monoclinic phase rapidly starts to form and, after 1000 hours, it reaches  $18.0 \text{ wt.}\%$ . This is observed both in

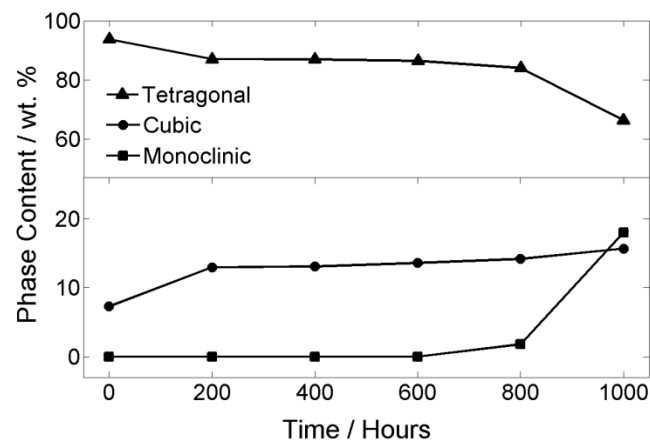


Figure 4. Tetragonal, cubic and monoclinic phase content in APS-deposited TC as a function of exposure time at  $1100^\circ\text{C}$ . Measurements performed at room temperature, after the heat treatments.

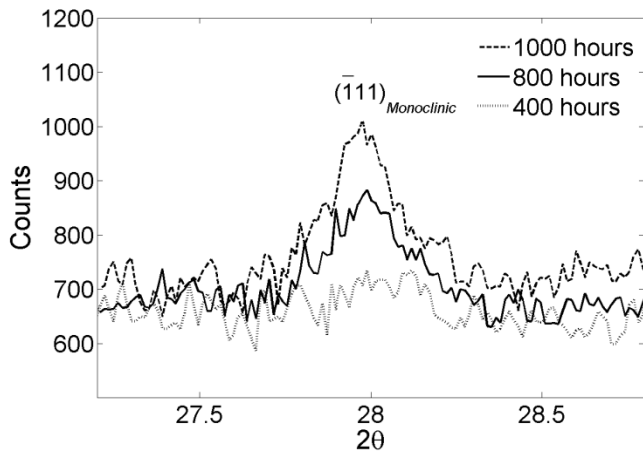


Figure 5. X-Ray diffractograms of TC treated at 1100°C for different exposure times. The peak corresponding to monoclinic phase first appears in samples treated for 800 hours. Measurements were performed at room temperature, after the heat treatments.

Fig. 4 and in the X-Ray diffractogram in Fig. 5. The uncertainty in the phase content was determined using the standard deviation. The uncertainty value was around 0.38 wt.% with a maximum of 0.43 wt.%

Fig. 4 also shows that the cubic phase forms in the first 200 hours at the expense of the tetragonal phase. Then, the cubic phase growth proceeds at a slower rate. After 800 hours, the monoclinic phase increases quickly, overpassing the cubic content before 1000 hours.

Fig. 6 shows how the Yttrium content of the metastable  $t'$  phase at room temperature reduces with exposure time at 1100°C. It is known that the  $t'$  phase decomposes into a mixture of stable tetragonal and cubic phases [37] as a consequence of Yttrium diffusion. However, for crystallographic purposes, both tetragonal structures can be analyzed as the same tetragonal polymorph in the zirconia solid solution [28,38].

As can be observed in Fig. 6, the  $Y_2O_3$  content constantly decreases in the tetragonal ( $t'$ ) phase with exposure time. After 800 hours, the  $Y_2O_3$  content in  $t'$  phase decreases to 6.60 wt.%. Then, the Y-depleted tetragonal phase transforms to a monoclinic phase during cooling and the monoclinic phase becomes more stable, favored by the Yttrium reduction in the tetragonal phase. Another factor which promotes the tetragonal-to-monoclinic phase transformation is the grain size [36,39,40]. From thermodynamic formulations, some researchers [39] have found that the surface energy of the tetragonal phase is lower than that of the monoclinic phase for a grain size smaller than 200 nm. Then, for a grain size smaller than 200 nm, the tetragonal phase is more stable. In addition, greater grain sizes favor the diffusion rate through the grain boundaries [36]. Therefore, it can be concluded that the Yttrium diffusion from the tetragonal phase increases and the amount of monoclinic phase formed from the Yttrium-depleted tetragonal phase increases.

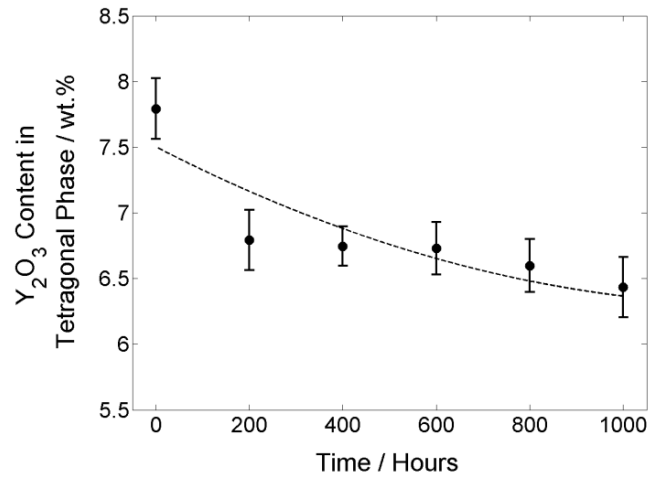


Figure 6.  $Y_2O_3$  content in the tetragonal phase ( $t'$ ) at room temperature as a function of exposure time at 1100°C.

### 3.2. Effect of the treatment temperature for fixed exposition time

The results of Rietveld measurements performed in the samples treated at different temperatures for 50 hours are presented in Fig. 7. The cubic phase increases slightly (around 3 wt.%) from 700°C to 1100°C. The uncertainty was around 0.41 wt.% with a maximum value of 0.44 wt.%. No monoclinic phase was detected in any treatments. Therefore, it can be said that the tetragonal phase decreases in the same proportion as the cubic phase increases. On the other hand, the cubic content increases with both temperature and exposure time. This behavior is in agreement with the results found in the literature in which the cubic content after 100 hours at 1200°C is around 19.0 wt.% and it reaches more than 40.0 wt.% for heat treatments at 1400°C after 100 hours [31].

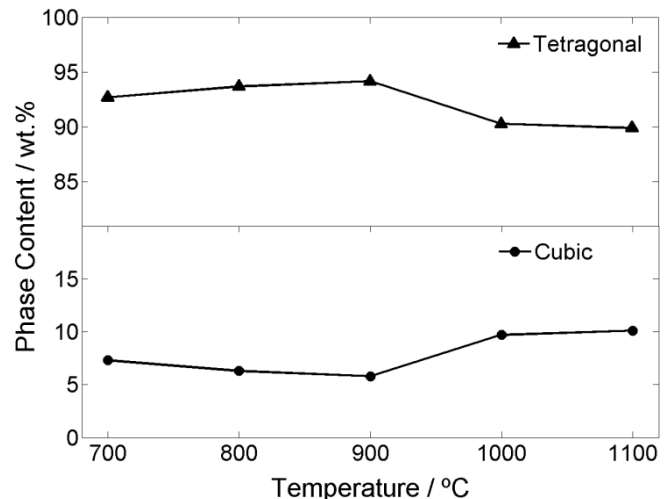


Figure 7. Tetragonal and cubic phase content in APS-TC as function of temperature after 50 hours of treatment. Measurements performed at room temperature, after the heat treatments.

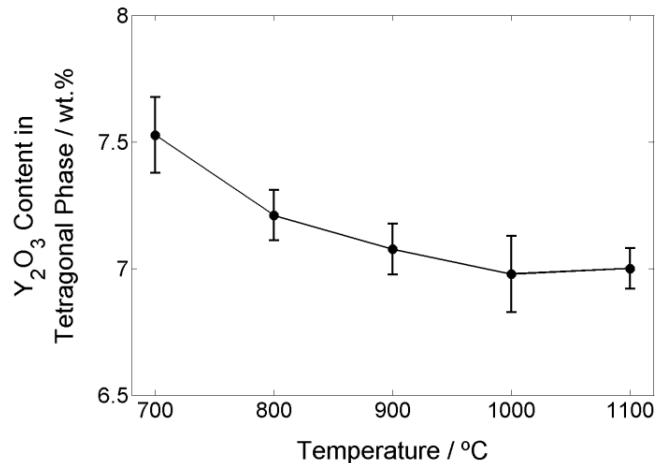


Figure 8. Y<sub>2</sub>O<sub>3</sub> content in the tetragonal phase as a function of temperature after 50 hours of treatment. Measurements performed at room temperature, after the heat treatments.

The effect of the temperature in the Y<sub>2</sub>O<sub>3</sub> content in the tetragonal phase is shown in Fig. 8. A slight reduction from 7.5 wt.% to around 7.0 wt.% is observed. On the other hand, the slight increment in cubic content is probably caused by the diffusion of the Yttrium from the tetragonal (*t'*) phase to stabilize the cubic phase. According to the results presented in Fig. 6, the monoclinic phase appears when the Y<sub>2</sub>O<sub>3</sub> content reduces to 6.6 wt.% or below. Therefore, it is not expected that the monoclinic phase forms after 50 hours at any temperature equal to or below 1100°C. The generality of the value of 6.6 wt.% of Y<sub>2</sub>O<sub>3</sub> in the tetragonal phase that was found in this work, at which the tetragonal phase destabilizes to transform in monoclinic phase during cooling requires additional research. Other factors, such as grain size and stresses, can favor the monoclinic stabilization.

#### 4. Conclusions

The phase transformations in APS-deposited TCs composed of ZrO<sub>2</sub> – 8 wt.% Y<sub>2</sub>O<sub>3</sub> under different heat treatment conditions were studied through XRD and Rietveld semi-quantitative measurements. The tetragonal structure (*t'*) generated from the APS deposition process became unstable at high temperatures. The increase in temperature and exposure time favored Yttrium diffusion from the tetragonal phase and promoted formation of the cubic phase. The amount of such a cubic phase increased from 7.3 wt.% at room temperature to 15.7 wt.% after 1000 hours at 1100°C. After 800 hours at 1100°C, the monoclinic phase started to form and the Y<sub>2</sub>O<sub>3</sub> content in the tetragonal phase reduced to values below 6.6 wt.%; the amount of the monoclinic phase increased rapidly and reached 18.0 wt.% after 1000 hours at this temperature.

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