Research Article

Hot Corrosion of SrTiO₃ Perovskite in Na₂SO₄ + 50 wt.% V₂O₅ and Na₂SO₄ + 10 wt.% NaCl Environments at 900°C

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This study examines the phase stability of perovskite SrTiO₃ in Na₂SO₄ + 50 wt.% V₂O₅ and Na₂SO₄ + 10 wt.% NaCl environments at 900°C. Hot corrosion results show the formation of Sr₂VO₄, SrV₂O₆, and SrTiV₅O₁₁ phases in Na₂SO₄ + 50 wt.% V₂O₅ environment and Sr₃Ti₂O₇, Na₄TiO₄, and TiO₂ phases in Na₂SO₄ + 10 wt.% NaCl environment. Morphological observations revealed the austerity of hot corrosion attack on SrTiO₃. The Sr²⁺ ions leached out from SrTiO₃ and reacted with corrosive environments. These observations clearly indicate the destabilization of SrTiO₃ in both environments.

1. Introduction

Thermal barrier coatings (TBCs) are generally applied as thermal insulating layers on the hot metallic surfaces such as combustion engine parts, gas turbine blades, and aero-engine parts. TBCs can enhance the operating temperature and reduce the cooling requirements ensuing higher engine efficiency and part life of an engine [1–5]. In order to gratify the requirements of TBC materials, the properties such as low thermal conductivity, higher melting point, and matching thermal expansion coefficient with bond coat are needed. Another predominant factor for determining the durability of TBCs is the stability against hot corrosion. Hot corrosion occurs when the engine runs with low quality fuel which contains sodium, vanadium, and sulfur as impurities in it and also in chloride (marine) environments. The sodium and vanadium salts are having lower melting point (700–880°C) than gas turbine operating temperature (>1300°C). Hence, their salts can easily condensate in the form of Na₂SO₄, V₂O₅, and NaCl on the top coat of TBCs and undergo hot corrosion

in two types (Types I and II). Type I generally occurs above the melting point of sodium sulphate (T_m ~ 884°C) with Type II vice versa. In both the types, these salts react with ceramic top coat and show the way for destabilization and degradation of TBCs.

In recent years, 6–8 wt.% yttria stabilized zirconia (YSZ) has been widely used as a top coat material for TBC and much research has been involved in understanding the hot corrosion behavior of YSZ in corrosive environments. The authors reported that these molten salts could cause several microcracks and spallation of the coating [6–9]. Particularly, in vanadium containing environments, YSZ is found to be unstable and forms YVO₄ and m'-ZrO₂ (destabilization). Destabilization causes the volume change and leads to spallation of the coating.

Therefore, there is a need to develop alternate materials to enhance the hot corrosion stability of the coating. The candidate materials, such as garnets (Y₃Al₅O₁₂), La, Sm, Nd, Yb, and Gd doped ZrO₂, YSZ-Ta₂O₅, monazite (LaPO₄), and lanthanum aluminates (LaMgAl₁₁O₁₉) were developed

in recent years and their stability was examined [10–14]. Perovskite (ABO_3) based ceramics (SrZrO_3 , SrCeO_3 , and BaZrO_3) have also drawn much attention towards TBCs due to their low thermal conductivity, higher melting point, and matching thermal expansion coefficient with bond coat material. Particularly, the SrTiO_3 has higher melting point of 2080°C , thermal expansion coefficient $\sim 10^{-5} \text{ K}^{-1}$ (1727°C), and thermal conductivity in the range of 11 to $2.5 \text{ W}\cdot\text{m}^{-1} \text{ K}^{-1}$ (25°C to 1100°C). But the review of literature showed that the published work on SrTiO_3 is mostly devoted to understanding the thermophysical properties [15–20]. However, the literature on hot corrosion behavior of SrTiO_3 is sparse and reactions between SrTiO_3 and molten melts remain unclear. Therefore, further studies are needed to understand the hot corrosion behavior of SrTiO_3 .

Hence, the present work is focused on studying the phase stability of perovskite type SrTiO_3 in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C .

2. Materials and Methods

2.1. Sample Preparation. Strontium titanate powders were synthesized via hydrothermal route [21] using strontium hydroxide (99%, Alfa Aesar) and titanium tetrachloride (99.9%, Fisher Scientific) as precursor materials. To make the pellets, obtained strontium titanate powders were pressed with 370 MPa pressure using a uniaxial die (1.3 cm internal diameter). Polyvinyl alcohol (PVA) was used as a binder (1:10 ratio) in the preparation of strontium titanate pellets. Compacted pellets were kept in a furnace at 500°C for 48 h to remove the binder. Then these pellets were heat treated at 900°C for 48 h.

2.2. Hot Corrosion Tests. A mixture of Na_2SO_4 and V_2O_5 salts in 1:1 weight ratio and another mixture of Na_2SO_4 and NaCl salts in 9:1 weight ratio were prepared and dissolved separately in distilled water for getting 50 wt.% of each salt solution. Samples were preheated at 200°C for 30 min to achieve good adhesion of salts. Salt solution was applied on all sides of surface using camel hair brush. Salt coated samples were kept in a furnace and heated at 120°C for 2 h to remove the moisture. The samples were weighed to determine the salt coverage and found in the range of $20\text{--}30 \text{ mg}/\text{cm}^2$. Then these salt coated samples were kept in an alumina boat and heated at 900°C for 100 h in air. After hot corrosion, samples were taken out and washed with running water and dried.

2.3. X-Ray Diffraction Analysis. Before and after hot corrosion, the nature of phases was investigated by X-ray diffractometer (Bruker, D8 advance) with $\text{Cu K}\alpha$ radiation with a scan rate of $6^\circ/\text{min}$. All the phases were identified using the JCPDS-PCPDFWIN software package.

2.4. Morphological Analysis. The surface morphologies and elemental analysis of samples were examined by Field Emission Scanning Electron Microscope (FESEM-ZEISS-SUPRA 55VP) attached with EDX (OXFORD instruments).

FIGURE 1: XRD patterns of the SrTiO_3 , (A) before exposure to hot corrosion and after exposure to hot corrosion at 900°C in (B) $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment and (C) $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment.

3. Results

3.1. X-Ray Diffraction Analysis. Figure 1 shows the X-ray diffraction patterns of the SrTiO_3 sample (A) before hot corrosion and after subjecting to hot corrosion at 900°C in (B) $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment and (C) $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment. Before hot corrosion, it is observed that SrTiO_3 is in cubic phase (JCPDS-79-0176). After exposure to hot corrosion in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment (Figure 1(B)), additional phases such as Sr_2VO_4 , SrV_2O_6 , and $\text{SrTiV}_5\text{O}_{11}$ (JCPDS-81-0854, 28-1267, and 48-0540, resp.) were identified. After exposure to hot corrosion in $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment (Figure 1(C)), additional phases such as $\text{Sr}_3\text{Ti}_2\text{O}_7$ (JCPDS-78-2479), Na_4TiO_4 (JCPDS-42-0513), and TiO_2 were identified. It is worthwhile to mention that SrTiO_3 phase is found to be unstable in both $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C . Succinctly, this indicates destabilization of SrTiO_3 .

3.2. Morphological and Microchemical Analysis. Surface morphology of SrTiO_3 sample before and after exposure to hot corrosion in both $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C was examined to identify the severity of hot corrosion attack. Figure 2 shows the macroscopic view of the SrTiO_3 before and after exposure to hot corrosion. Figure 2(a) (before hot corrosion) reveals that surface is free from cracks and scales. Nonuniform and severe hot corrosion attack was noticed on the surfaces which were subjected to hot corrosion in both $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ (Figure 2(b)) and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments (Figure 2(c)) at 900°C .

Figure 3 shows the surface morphology of SrTiO_3 before subjecting to hot corrosion. Figures 3(a) and 3(b) show the

FIGURE 2: Macroscopic view of SrTiO₃ surface, (a) before exposure to hot corrosion and after exposure to (b) Na₂SO₄ + 50 wt.% V₂O₅ environment and (c) Na₂SO₄ + 10 wt.% NaCl environment at 900°C.

FIGURE 3: (a) Lower magnification image of SrTiO₃ sample before exposure to hot corrosion and (b) higher magnification image of (a). EDX analysis was done on the surface of (b) at region 1 (corresponding to SrTiO₃).

TABLE 1: The elemental compositions of SrTiO_3 samples before and after being subjected to hot corrosion in both $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C .

S. No	Figures	Environments	Regions	Elements (wt.%)			
				O	Ti	V	Sr
1	Figure 3(b)	Before hot corrosion	1	24.80	22.82	-	52.38
2	Figure 4(b)	$\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$	1	24.94	23.14	-	51.92
3	Figure 4(c)	$\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$	1	25.03	-	18.83	56.14
4	Figure 6(b)	$\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$	1	26.90	27.92	-	45.18

FIGURE 4: Surface morphology of SrTiO_3 after being subjected to hot corrosion in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment at 900°C , (a and b) lower and higher magnification images of SrTiO_3 , shows octahedron shaped morphology. EDX analysis was demonstrated on (b) at region 1 (corresponding to strontium titanate); (c) higher magnification image at other regions of (a) shows irregular shape morphology. EDX analysis was done on (c) at region 1 (corresponding to strontium vanadates).

lower and higher magnification FESEM images of SrTiO_3 . Figure 3(b) shows the spherical shape morphology. EDX analysis was performed to identify the chemical compositions at region 1 (Figure 3(b)) and the data is presented in Table 1. Region 1 is composed of Sr, Ti, and O (corresponding to SrTiO_3). These results are in good agreement with the XRD results (Figure 1(A)). The surface morphology of SrTiO_3 after subjecting to hot corrosion in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment at 900°C reveals the octahedron shape morphology (Figure 4(a)). Higher magnification image of Figure 4(a) is shown in Figure 4(b) and EDX analysis was done at region 1 (data is presented in Table 1). Region 1 is composed of Sr, Ti, and O (corresponding to SrTiO_3). Spherical type of morphology of strontium titanate changes onto octahedral shape when treated in sulphate solution of vanadate at 900°C .

Figure 4(c) shows irregular shape morphology. EDX point analysis was carried out at region 1 (data is presented in Table 1) and this location is composed of Sr, V, and O (corresponding to strontium vanadates). Figure 5 shows the severity of hot corrosion on SrTiO_3 after exposure to $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment and it shows the porous layer and corrosion products on the surface of SrTiO_3 .

The surface morphology of SrTiO_3 after exposure to $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C is shown in Figure 6. Lower magnification image (Figure 6(a)) reveals that nonuniform, severe hot corrosion and groove attack were observed in $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment. Higher magnification image (Figure 6(b)) reveals the plate type of morphology. EDX analysis was carried out at region 1 (data is presented in Table 1) in Figure 6(b) and this location is

FIGURE 5: Surface morphology of SrTiO₃ after being subjected to hot corrosion in Na₂SO₄ + 50 wt.% V₂O₅ environment at 900°C. A porous layer and corrosion products were observed on the surface.

composed of Sr, Ti, and O (corresponding to Sr₃Ti₂O₇). Similar type of morphology for Sr₃Ti₂O₇ has also been observed by previous author [22].

4. Discussion

The phase stability of SrTiO₃ against hot corrosion is being studied in Na₂SO₄ + 50 wt.% V₂O₅ and Na₂SO₄ + 10 wt.% NaCl environments at 900°C. Though SrTiO₃ is an oxide, it underwent destabilization in both environments. Massive hot corrosion attack is being observed on surface of SrTiO₃ after being subjected to both environments at 900°C. Significant literature is available for SrO-V₂O₅ binary systems but not for SrO-NaVO₃. It is reasonable to predict the chemical reaction between SrTiO₃ and NaVO₃ using SrO-V₂O₅ [23, 24] and SrO-VO₂-V₂O₅ phase diagrams [25]. First, SrTiO₃ decomposed into SrO and TiO₂ phases by leaching of Sr²⁺ ions from SrTiO₃ due to the presence of corrosive environments (see (1)). When the temperature reaches 630°C in furnace, NaVO₃ forms and acts as good solvent medium for oxides. At this stage, the reactions between sodium vanadate and oxides are faster.

Habibi et al. [14] have also reported that the liquid NaVO₃ will increase the atom mobility of oxide elements. SrO reacts with NaVO₃ liquid melt and forms SrV₂O₆ (see (2)). Again Na₂O reacts separately with V₂O₅ and SO₃ and forms NaVO₃ and Na₂SO₄, respectively. This reaction is cyclic. SrO and TiO₂ can also react with NaVO₃ and form SrTiV₅O₁₁. At 900°C, SrO reacts with NaVO₃ and forms the Sr₂VO₄ phase (see (3)). This phase is identified as predominant corrosion product phase in XRD (Figure 1(B)). The above sequence of reactions describes the formation mechanism of corrosion products. Higher magnification image of SrTiO₃

(Na₂SO₄ + 50 wt.% V₂O₅ at 900°C) (Figure 5) shows the severe hot corrosion and corrosion products on it. Due to damage caused by Na₂SO₄ + 50 wt.% V₂O₅, a porous layer and corrosion products were formed and observed in the Figure 5. This reveals the severity of hot corrosion on SrTiO₃ in Na₂SO₄ + 50 wt.% V₂O₅ environment.

The Na₂SO₄ + 10 wt.% NaCl environment also caused the leaching Sr²⁺ ions and subsequently formation of SrO and reacts with chloride to form SrCl₂ (see (4)). In general thermodynamic stability of oxides is higher than the chlorides. Hence formed chlorides are further transformed into oxides (see (5)). Further they reacted with themselves and formed Sr₃Ti₂O₇ at 900°C (see (6)). Tilley [26] has studied the Sr-Ti-O system at 1100–1473°C and reported the formation of three phases such as Sr₂TiO₄, Sr₃Ti₂O₇, and SrTiO₃. Jacob and Rajitha [27] have studied the pseudo-binary system (SrO + TiO₂) at 775 to 977°C and reported the thermodynamics properties of Sr₂TiO₄, Sr₃Ti₂O₇, Sr₄Ti₃O₁₀, and SrTiO₃ using solid state galvanic cells (see (6)). Based on the previous works [22, 27] the reaction between SrO and TiO₂ can be expressed as follows:

The TiO₂ phase can react with Na₂O to form Na₄TiO₄ (see (7)). There has been no previous work on hot corrosion behavior of SrTiO₃ in NaCl medium. The possible role of Na₂O against SrO and TiO₂ phases is discussed here. According to Coulomb's law (lattice energy depends on the ionic radii and charges of the ions) the compounds with +2 ions have more lattice energy than the compounds with +1 ion. In addition to this, Ti atoms are having smaller ionic radius (94 pm) than that of Sr atoms (112 pm) [28]. Hence, the Ti-O structured atoms would be expected to have larger lattice energy which gives higher attractive force towards Na₂O. Based on our above discussions the order of lattice energy can be expressed as

The lattice energy of TiO₂ (12150 kJ/mole) is more than that of SrO (3223 kJ/mole) [29]. As per theory, the lattice energy is directly proportional to the phase stability of the compound, which indicates the less stability for SrO at 900°C. But, in the case of TiO₂, it needs higher energy to separate the Ti²⁺ and O²⁻ ions. As we discussed earlier, the strong electrical attraction between the Na⁺ (Na₂O) and TiO₂ ions might cause the formation of Na₄TiO₄ phase (see (7)). These discussions are well in agreement with XRD (Figure 1(C)) results. This is the possible reason why the reaction between TiO₂ and NaCl was highly favored compared with that with SrO. According to the binary phase diagrams of Na₂O-TiO₂ [30, 31], Na₄TiO₄ melts at 850°C and this was the reason

FIGURE 6: (a) Surface morphology of SrTiO_3 after exposure to hot corrosion in $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment at 900°C and (b) higher magnification image of (a) shows the plate type morphology. EDX analysis was performed on (b) at region 1 (corresponding to $\text{Sr}_3\text{Ti}_2\text{O}_7$).

for Na_4TiO_4 being identified as minor corrosion product compared to that of $\text{Sr}_3\text{Ti}_2\text{O}_7$ in XRD (Figure 1(C)).

5. Conclusions

The hot corrosion behavior of perovskite type SrTiO_3 was studied in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C for 100 h.

- (1) SrTiO_3 has been found to be unstable in both $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ and $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environments at 900°C .
- (2) The Sr^{2+} ion leaches from its own phase and reacts with NaVO_3 to form Sr_2VO_4 , SrV_2O_6 and $\text{SrTiV}_5\text{O}_{11}$ in $\text{Na}_2\text{SO}_4 + 50 \text{ wt.}\% \text{ V}_2\text{O}_5$ environment.
- (3) The $\text{Na}_2\text{SO}_4 + 10 \text{ wt.}\% \text{ NaCl}$ environment also caused the leaching of Sr^{2+} ions. The SrO reacted with TiO_2 and formed $\text{Sr}_3\text{Ti}_2\text{O}_7$. The reaction between Na_2O and TiO_2 caused the formation of Na_4TiO_4 .
- (4) This study clearly indicates the destabilization of SrTiO_3 in both mediums.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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