Effect of magnesium addition on the structural, microstructural and electrical properties of YVO₄

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ABSTRACT

The synthesis of Y₁₋ₓ/₂MₓV₁₋ₓ/₂O₄₋ₓ/₂ (MgₓYV) (x = 0.0–0.5) by solid state reaction technique is reported. This preparation method is compared with chemical synthesis method such as nitrate-citrate auto-combustion route. The crystal structure and the surface morphology of the samples were determined by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques, respectively. Substantial decrease in phase formation temperature was observed for combustion method. The solubility limit of Mg in YVO₄ lattice was found to be low i.e., x ≤ 0.1. The temperature dependence of conductivity of MgₓYV samples indicated an increase in ionic conductivity values compared to the parent YVO₄ due to formation of oxygen ion vacancies produced by the incorporation of acceptor Mg cations. Combustion-synthesized MgₓYV samples showed higher conductivity compared to the samples prepared by conventional solid state reaction.

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1. Introduction

Over the past few decades, considerable research efforts have gone into the development of ionically conducting solids, especially oxygen ion conductors. The importance of these materials has generated considerable scientific and technological interest and there have been a continued drive for their application in vital areas such as solid electrolytes in solid oxide fuel cells, oxygen pumps, gas sensors, oxygen permeable catalysts, batteries, electrochromics such as solid electrolytes in solid oxide fuel cells, oxygen pumps, counter electrodes in electrochromic devices, gas sensors, components of oxide fuel cell anodes, oxidative dehydrogenation of light alkanes etc [10–12]. As for the host lattice, yttrium orthovanadate (YVO₄) has been considered to be an attractive candidate for incorporation of several metal ions [13]. YVO₄:Eu²⁺ is known to be a typical commercial red emitting phosphor which can be used in several places such as in cathode ray tubes, fluorescent lamps, X-ray detectors etc [14,15]. The conductivity of zircon-type CeVO₄ systems are well studied with different dopants [12,16]. Oxygen ion conductivity of zircon type Ce₁₋ₓAₓVO₄₋ₓ (A = Ca, Sr; x = 0–0.2) was discussed by Tsipis et al. [12]. They showed that the unusual behavior of conductivity is due to the presence of oxygen deficient CeO₂ separation while increasing the temperature to 1100 °C and also reported that oxygen ionic conductivity is independent of A-
site dopant content.

To date, the ionic conductivity of YVO₄ still has not been studied and reported. In this context, we have chosen YVO₄ as our parent material and explored the effect of substitution by alkaline earth ion such as magnesium on the structural, microstructural and electrical properties. The relatively good stability of Mg²⁺ allows for a study of its effects on the electrical and dielectric properties without the reduction of the substituent ion[17]. Many reports are available on the studies of acceptor-type Mg substitution on different cationic sites aiming to improve the transport properties[18–20]. Kinoshita et al.[19] studied the effect of Mg substitution on the oxide ion conductivity of apatite-type lanthanum silicate (La₀.₃₃Si₆O₂₆). According to them the partial replacement of La by Mg decreased the conductivity, whereas substitution for Si increased the conductivity with the amount of Mg and dual doping resulted in slight improvement of conductivity. Recently, Li et al.[20] reported a new oxide ion conductor based on ferroelectric perovskite Na₀.₅Bi₀.₅TiO₃. Mg doping at the Ti site of this system improved the electrolyte stability in reducing atmospheres, lowered the sintering temperature and also showed a large increase in the ionic conductivity.

The present work was aimed to study the effect of the addition of magnesium as a third cation into the host lattice of a tetragonal zircon type yttrium orthovanadate. The work was focused on the preparation of a new mixed ceramic oxide, viz; magnesium yttrium vanadate (Y₁₋ₓ/₂MgxV₁₋ₓ/₂O₄₋δ, x = 0 – 0.5), via two synthetic procedures and a comparative study of their structural, microstructural and electrical properties.

2. Experimental

Yttrium Vanadium oxide compounds (YVO₄) with different quantities of magnesium were prepared using solid state synthesis and combustion synthesis. The synthesized compositions are designated as YV and MgxYV, where YV stands for YVO₄ and MgxYV for Y₁₋ₓ/₂MgxV₁₋ₓ/₂O₄₋δ; and the numeral following Mg represents the particular amount of magnesium (x) in the Y₁₋ₓ/₂MgxV₁₋ₓ/₂O₄₋δ (x = 0 – 0.5) formula. The synthesis method adopted is abbreviated as SS for solid state synthesis and CS for combustion synthesis.

2.1. Solid state synthesis (SS)

The MgₓYV (x = 0.0–0.5) samples were prepared through conventional solid state reaction method using high-purity magnesium carbonate hydrate (Mg₅(CO₃)₄(OH)₂. 5H₂O, 99.0%, Sigma–Aldrich, St. Louis, MO, USA), yttrium oxide (Y₂O₃, 99.0%, IRE, India) and vanadium pentoxide (V₂O₅, 99.6%, Sigma–Aldrich) as the starting materials. Stoichiometric amount of the starting materials were weighed accurately, and mixed manually in acetone medium using agate mortar and pestle. The resultant mixture was then first calcined at 650 °C for 6 h followed by calcinations at different temperatures (750–1000 °C/h) with intermediate grinding. These powders were then ground well and mixed with 4 wt% polyvinyl alcohol, PVA (molecular weight 72,000, BDH Lab suppliers, Poole, U.K), dried and ground well. Cylindrical disks of ~11 mm diameter and ~2–3 mm thickness were prepared by applying a pressure of 100 MPa by a uniaxial press. These cylindrical compacts were then sintered at 1250 °C/6 h with an intermediate soaking time of 600 °C to expel the binder PVA.

2.2. Combustion synthesis (CS)

A wet chemical synthetic route viz., Pechini process was adopted for the preparation of MgₓYV (x = 0–0.5). Samples were synthesized using magnesium nitrate hexahydrate (Mg(NO₃)₂. 6H₂O, 98.0% from Alfa Aesar, UK), citric acid anhydrous (C₆H₈O₇, 99.5%, Merck specialties private limited, India), yttrium oxide (Y₂O₃, 99.0%,

Fig. 1. Powder X-ray pattern for M₀.₁YV synthesized by (a) SS (b) CS method.

Fig. 2. Variation of density as a function of temperature for M₀.₁YV synthesized by SS and CS method.
IRE, India), ammonium metavanadate (NH$_4$VO$_3$, 99.0%, Sigma-Aldrich, USA), and ethylene glycol (C$_2$H$_6$O$_2$, >99.0%, Merck, India) as the starting materials. Y$_2$O$_3$ was dissolved in nitric acid, and aqueous solutions of Mg(NO$_3$)$_2$.6H$_2$O and NH$_4$VO$_3$ were added to it. Citric acid was added to resulting aqueous solution. The solution was then heated to about 100 °C under constant magnetic stirring, and ethylene glycol was added to this for esterification. During the synthesis, the solution underwent distinctive color change at different stages which was attributed to the change in oxidation states of vanadium ions. Finally, a light yellow color occurred [21]. The solution was further heated to about 300 °C. During this heating the solvent evaporated and a polymeric resin containing the metal ions was formed. Subsequently this polymerized complex underwent auto ignition leading to the formation of voluminous fluffy powder with a dark color. This powder was then ground well and was calcined at various temperatures in the range 550 °C—1100 °C in air for 6 h. The calcined powders were then ground well, mixed with PVA solution, dried and uniaxially pressed into cylindrical discs of about 11 mm diameter and around 2–3 mm thickness and sintered at 1200 °C/6 h in air.

The formation of required phase in the as obtained, calcined and sintered samples was studied at room temperature using X-ray powder diffractometer (X’pert PRO, PANalytical, Netherlands), using Ni filtered Cu Kα radiation. The coefficient of thermal expansion (CTE) was measured using thermomechanical analyzer (TMA/SS7300, SII Nano Technology Inc.). The microstructures of the polished and thermal etched compacts coated with palladium-gold mixture were studied using a scanning electron microscope (SEM) (JEOL-JSM, 5600 LV, Tokyo, Japan). Microstructural characterization along with elemental mapping of Mg$_{0.1}$YV was performed by scanning electron microscopy (Hitachi SU-70) coupled with energy dispersive spectroscopy (EDS) (Bruker, Quantax 400detector). Particle shape and size of the samples prepared by CS method were studied using high resolution transmission electron microscopy (TEM) (JEOL-JSM, 5600 LV, Tokyo, Japan).
microscope (HRTEM) (Tecnai G2, FEI, Netherlands). Powder samples calcined at 550 °C were finely ground and dispersed in acetone by ultrasonication. A drop of this was cast onto carbon-coated copper grids for the TEM analysis. The densities of the sintered samples were obtained by Archimedes method using deionized water as the immersing medium. The oxidation state of vanadium was determined using electron paramagnetic resonance (EPR). The spectra of the samples were recorded using EPR spectrometer (Bruker EMX plus EPR spectrometer, Germany) operating at room temperature and at 9.87 GHz with 100.0 kHz field modulation. The electrical conductivity ($\sigma$) was determined by impedance spectroscopy (Agilent 4284A precision LCR meter, frequency range 20 Hz–1 MHz) using disk-shaped sintered samples (thickness ~ 2 mm, $\phi$ ~ 10 mm) with porous Pt electrodes applied onto flat surfaces. The measurements were performed as function of temperature at 500–900 °C in air.

3. Results and discussion

3.1. Phase analysis and densification

X-ray diffraction study was primarily used to analyze the phase purity and crystal structure of the samples. The XRD pattern for M0.1YV (SS) and M0.1YV (CS) powder prepared by the two synthetic methods at different temperatures is given in Fig. 1.

Fig. 1(a) represents the XRD pattern for SS M0.1YV samples heat-treated in a temperature range of 800 °C–1200 °C/6 h. Complete phase formation is clearly visible starting from 1000 °C. All the peaks can be indexed using tetragonal $I4_1/amd$ space group characteristic for zircon-type YVO$_4$ (JCPDS No.82-1968). X-ray pattern of CS M0.1YV (Fig. 1(b)) consisted of amorphous like phases at lower temperatures. The XRD pattern obtained after calcination at 550 °C showed the presence of zircon structured compound and no other crystalline phases coexisted, indicating the formation of a single phase material. Improved crystallinity is observed after calcinations at temperatures over 650 °C. In this method metal ions are uniformly distributed in the polymeric resin and gives molecular level

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Crystallographic refinement parameters for Y$<em>{1-x/2}$Mg$<em>x$V$</em>{1-x/2}$O$</em>{4+x/2}$ ($x$ = 0.0 and 0.1).</th>
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<tr>
<td>Crystallographic parameters</td>
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<td>$R_{exp}$%</td>
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<tr>
<td>GOF</td>
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</table>

Fig. 5. Rietveld refined XRD data for M0.1YV (SS).

Fig. 6. EPR spectrum of M0.1YV (SS) at room temperature.
mixing of the elements, leading to the formation of the desired phase at lower temperatures compared to solid state route [22]. Fig. 2 shows the density variation for M₀.₁YV heated at different temperatures between 800 °C/6 h to 1250 °C/6 h and 800 °C/6 h to 1200 °C/6 h for SS and CS samples respectively. For SS, the maximum density was obtained for the samples sintered at 1250 °C/6 h whereas for combustion synthesized it was 1200 °C/6 h. In both the cases, the materials showed similar trend with increasing temperature. Hence, temperature - time condition for optimum sintering was taken as 1250 °C and 6 h duration for SS and 1200 °C and 6 h for CS samples. Maximum relative density obtained for SS and CS samples were 96.9% and 93.7% respectively. The slightly lower values of density for the CS samples compared to the SS may be due to the presence of porosity arising because of the agglomerate formation by the smaller particles obtained in the former case. It is confirmed by the TEM as well as the SEM analysis shown in Figs. 3, 7 and 8.

3.2. TEM analysis

Fig. 3 shows the TEM micrographs of powder samples of MgₓYV (CS) calcined at 550 °C/6 h. The images showed grains of spherical...
morphology, with an average particle size below 50 nm. The powder samples were found to be agglomerated together even during the combustion reactions [23].

3.3. Structural studies

Fig. 4 demonstrates the XRD patterns of MgxYV prepared by SS method. The patterns for the samples prepared by SS and CS methods show single-phase compositions up to x = 0.1, and can be completely indexed with JCPDS No.82-1968. However, as the x value increased from 0.2 to 0.5 a minor impurity peak was observed at 2θ value of around 42°. This peak in the XRD patterns was attributed to the presence of MgO (periclase) secondary phase, and the peak intensity increased with increasing x in Y1-x/2MgxV1-x/2O4-

During the refinement process, oxygen was always considered with full occupancies, whereas yttrium, magnesium and vanadium occupancies were varied in accordance with the stoichiometry. Refinement parameters obtained for YV and Mg0.1YV are listed in Table 1. The unit cell volume as well as the lattice parameters was found to decrease on substitution. This can be a consequence of insertion of Mg ions in to the lattice.

In fact, there is a considerable mismatch of ionic sizes (ionic radii: Y³⁺ (CN8) = 1.019 Å; V⁵⁺ (CN4) = 0.355 Å; V⁴⁺ (CN4) = 0.45 Å (extrapolated); Mg²⁺ (CN4) = 0.57 Å; and Mg²⁺ (CN8) = 0.89 Å [24]), so the solubility limit of Mg cations in either sublattice is expected to be limited. As per the XRD analysis (Fig. 4), the solid solution formation of the MgxYV samples is found to be x ≤ 0.1. The possible mechanisms of Mg incorporation into crystal lattice of YVO4 may include:

\[
xMgO \rightarrow YVO_4 \quad (Y_{1-0.5x}Mg_{0.5x})(V_{1-0.5x}Mg_{0.5x})O_4-x
\]

\[
xMgO \rightarrow Y_{1-x}Mg_xVO_4-0.5x + 0.5xY_2O_3
\]

\[
xMgO \rightarrow YV_{1-x}Mg_xO_4-1.5x + 0.5xV_2O_5
\]

or their combination, or segregation of some other impurities. As mentioned above, the results of refinement indicate the preference for the first mechanism within the solid solubility domain.

3.4. Structure refinement

Rietveld refinement of the solid state-synthesized YVO4 and Mg-doped samples were carried out using X’pert Highscore Plus software. Refinement pattern obtained for M0.1YV-SS sample is shown in Fig. 5. The reported tetragonal zircon structure of YVO4 with space group I41/amd (141) was used as a starting model for the refinement. Here Y ions are at (4a: 0, ¼, ½) sites, V ions at (4b: 0, ¼, ½) sites and O is at (16 h: x, y, z). Mg ions are assumed to be distributed either in 4a or in 4b sites or between both 4a and 4b sites. Refinement of all the 3 possibilities was carried out. All of these gave results in the acceptable range. The model with Mg ion distribution in both sites (4a and 4b sites) produced a better fit.
3.5. EPR analysis

Electron paramagnetic resonance (EPR) spectroscopy is useful technique for getting information about the nuclearity, elemental composition, and electronic structure of a paramagnetic center [25]. Room temperature EPR spectra of solid state synthesized MgₓYV consist of 8 lines with g value centered at ~1.98 and are due to V (IV) ion. Vanadium ions can have a wide range of oxidation states and among them most commonly encountered are V (IV) and V (V) states. V (V) having an outer electronic configuration 3p⁶3d⁰ which is diamagnetic in nature and EPR inactive, whereas the V (IV) has one unpaired electron in d orbital (3p⁶3d¹) is paramagnetic and EPR active. The strong absorption line seen in the spectra originates from the V (IV) paramagnetic center, since the other ions present in the systems are diamagnetic in nature and will not contribute to the EPR signals. The absorption lines arise due to the interaction of unpaired 3d¹ electron of V (IV) with the electromagnetic field in the microwave region. The existence of even a small number of paramagnetic centers should have a strong effect on the physical properties of the system which is in turn dependent on the high temperature treatment and also on the oxygen deficiencies. The reduction of V (V) may occur in the course of heat treatment as a result of oxygen losses from the lattice. The intensity of the EPR signal is an indicator of oxygen loss and has a direct relationship. The release of oxygen or the electron addition is needed for the reduction of V (V) [26]. A typical EPR spectrum obtained for MgₓYV (x = 0.5) is shown in Fig. 6. EPR studies have shown that Mg incorporation to YV lead to the reduction of V (V) to V (IV).

3.6. Surface morphology

In order to understand the microstructure of the samples, the sintered compacts were polished well and then thermally etched for 1 h at temperature 50°C below the sintering temperature. The microstructures of the thermally etched MgₓYV samples are shown in Figs. 7 and 8. The grain sizes of all the samples were in the size range between ~2 μm and ~7 μm for CS and ~2 μm – 10 μm for SS samples. In both cases, Mg doping improved the grain growth to some extent under identical processing/sintering conditions. EDX analysis indicates the presence of some grains which are Mg enriched from x ≥ 0.2 onwards. This confirms the presence of secondary phase (periclase) present in these samples, as indicated by the XRD data presented in Fig. 4.

The results of SEM/EDS analysis of fractured surface of M0.1YV Fig. 11. Examples of the impedance spectra of MgₓYV ceramics collected in air at 850°C. Estimated capacitance values correspond to (4–8) x 10¹² F/cm for the high-frequency process and increase with doping from ~3 x 10¹² to ~10¹³ F/cm for the low-frequency arc.

Table 2

<table>
<thead>
<tr>
<th>Composition</th>
<th>Temperature range (°C)</th>
<th>Eₐ (kJmol⁻¹)</th>
</tr>
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<tbody>
<tr>
<td>YV (SS)</td>
<td>580–900</td>
<td>128.9 ± 1.6</td>
</tr>
<tr>
<td>Mg₀.₁YV (SS)</td>
<td>500–900</td>
<td>82.1 ± 0.4</td>
</tr>
<tr>
<td>Mg₀.₂YV (SS)</td>
<td>500–900</td>
<td>86.8 ± 1.1</td>
</tr>
<tr>
<td>Mg₀.₃YV (SS)</td>
<td>500–900</td>
<td>88.1 ± 0.3</td>
</tr>
<tr>
<td>Mg₀.₁YV (CS)</td>
<td>700–900</td>
<td>77.0 ± 0.1</td>
</tr>
<tr>
<td>Mg₀.₀₅YV (CS)</td>
<td>500–900</td>
<td>81.8 ± 0.4</td>
</tr>
<tr>
<td>Mg₀.₁₀YV (CS)</td>
<td>500–900</td>
<td>95.4 ± 1.3</td>
</tr>
</tbody>
</table>

Note: The activation energy was calculated using Arrhenius model
\[\sigma = (A_0/T) \exp(-E_a/(RT))\]; given errors are standard errors.
phases may include MgO and a phase of Mg.

Based on the XRD, SEM/EDS and EPR observations, one may conclude that magnesium substitutes preferably into vanadium sublattices, and, for $x = 0.1$, this is accompanied by a minor segregation of impurities which are not detectable by XRD. The impurity phases may include MgO and a phase of Mg–V–O system.

3.7. Density measurements

The collation of density of the sintered $\text{Mg}_x\text{YV}$ ($x = 0.0–0.5$) samples prepared through both processes is presented in Fig. 10(a) and their corresponding relative density in Fig. 10(b). Density shows a decreasing trend with increase in value of $x$ due to the incorporation of magnesium. However, their sinterability increases as indicated by the increase in relative density (Fig. 10(b)). Even though there is only a slight difference in the sintering temperature ($1250 ^\circ C/6$ h for SS and $1200 ^\circ C/6$ h for CS); the densities of the samples are almost similar except for the parent $\text{YVO}_4$. The combustion-synthesized $\text{YVO}_4$ sample show somewhat lower density compared to $\text{YVO}_4$ prepared by solid state route, and this may be due to the highly agglomerated nature of the powders.

3.8. Electrical conductivity

Fig. 11 shows typical impedance spectra of $\text{Mg}_x\text{YV}$ ceramics. The spectra comprised two signals: high-frequency semicircle corresponding to the bulk resistance (contributed by secondary phases) and low-frequency arc assigned to the electrode process. No grain boundary contribution could be distinguished in the studied temperature range. Undoped $\text{YVO}_4$ is a semiconductor with comparatively low electrical conductivity in air, $\sim 10^{-5}$ S/cm at 900 °C (Fig. 12), and activation energy of 129 kJ/mol in the studied temperature range (Table 2). Although the nature of electrical transport in this material was never reported, the presence of electrode response in the impedance spectrum (Fig. 11) implies an ionic contribution to the total electrical conductivity. Minor doping with magnesium results in an increase of electrical conductivity of $\text{Mg}_{0.1}\text{YV}$ compared to the parent material, $\sim$ one order of magnitude at 900 °C, accompanied with a decrease of activation energy to 77–82 kJ/mol at 500–900 °C. Whatever the mechanism of substitution, incorporation of magnesium cations into either yttrium or vanadium sublattices (or both) should be compensated by formation of oxygen vacancies thus promoting oxygen-ionic transport. The effect of acceptor-type doping on electrical properties of $\text{YVO}_4$ is therefore similar to that reported to calcium-substituted $\text{LaVO}_4$ [27]. In both cases, however, the solubility of acceptor-type cations is very limited leading to only a moderate improvement of ionic conductivity (Fig. 12).

Fig. 14 shows typical CTE curves for $\text{Mg}_x\text{YV}$ (SS and CS) samples in air. The role of phase impurities is further emphasized by the data shown in Fig. 13. $\text{Mg}_{0.1}\text{YV}$ samples synthesized by combustion method were found to exhibit higher conductivity compared to their counterparts prepared by conventional solid state synthesis.
This can be attributed most likely to better homogeneity and more uniform distribution of dopant cations in the host structure of CS samples. Magnesium doping beyond x = 0.1 resulted in progressive segregation of insulating impurity (MgO) and decline of the total electrical conductivity of Mg0.9V ceramics.

3.9. Thermal expansion

Fig. 14 demonstrates the variation of coefficient of linear thermal expansion (CTE) of M0.9YV prepared by both synthetic methods from room temperature to 400 °C. The average thermal expansion values are 4.7 × 10⁻⁶/°C at 150–400 °C for M0.9YV (CS) and 3.8 × 10⁻⁶/°C for M0.9YV (SS) respectively. These values are in good agreement with the literature data on YVO₄ [30] and other zircon-type vanadates [12,28].

4. Conclusions

Y₁₋ₓMₓGe₂Vₓ₋₂₀₄₋₆ (x = 0.0–0.5) samples were prepared by adopting solid state reaction and combustion methods. Combustion synthesis showed zircon-type phase formation at lower temperatures compared to solid state method. XRD analysis indicated that single phase formation occurs up to x = 0.1 in both cases, whereas the results of SEM/EDS analysis suggest slightly lower solubility limit of Mg in the YVO₄ lattice. TEM analysis indicated the smaller grain size of CS samples calcined at 550 °C/6 h. EPR analysis indicated the presence of paramagnetic centers and which may be due to the partial reduction of V(V) ions. Electrical measurements carried out in air demonstrated that the ionic conductivities of all Mg-doped samples are higher than that of undoped YVO₄. M0.9YV-SS sample showed maximum conductivity (σ = 1.25 × 10⁻⁴ S/cm at 900 °C) among the prepared compositions due to its better homogeneity.

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