PREPARATION AND CHARACTERIZATION OF LANTHANUM ZIRCONATE TBC POWDERS FOR DIESEL ENGINE APPLICATION

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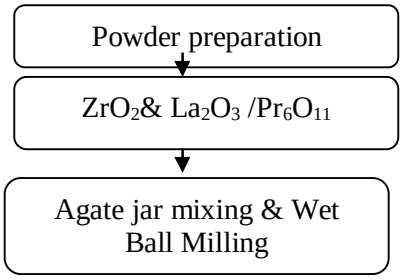
1.0.Introduction

Zirconia (ZrO_2) doped with oxides of Yytria, Calcia, Magnesia are well established systems for Thermal Barrier Coating (TBC) applications. TBC's are generally used in aero engines and in space vehicle components to protect them from the severe degradation arising due to high temperature environment.[1,2] Attempts are also being made to generate alternate and cost effective TBC's suitable for improving the energy efficiency of diesel engines in generators, heavy duty automotive vehicles etc. At the higher temperature (>1300 C), the well known 8% VPSZ TBC undergoes reversible phase transformation (t to m) due to which the coating becomes unstable[3,4]. To overcome this problem, we made an effort to develop Lanthanum Zirconate and La_2O_3 have been used as the dopant or stabilizer to zirconia powder as a candidate for TBC. Various characterization tests have been conducted such as XRD, EDAX etc. to optimize and determine the dopant concentration for partial or full stabilization of Zirconia. To study the effect of stabilization of Zirconia, two methods have been followed: Powder preparation by Agate jar mixing.,Powder preparation by Wet ball milling. In the first method, stabilization of Zirconia is very less due to higher particle size and higher calcination temperature of 1635^0C for four hours. In the second method, the particles are well grinded (5-microns) and calcined at 1450^0C for four hours. It is that 98% of Zirconate formed which is most suitable for the TBC applications

The preparation of plasma spray able grade powders consisted of the following steps as specified in the below chart.

2.1. Agate & Wet ball mill method



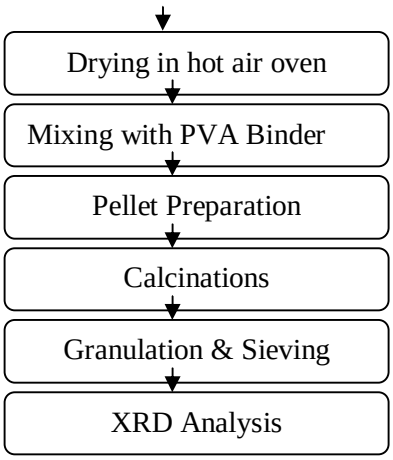


Fig1: Flow chart for $LA_2Zr_2O_7$ Powder Preparation by Wet Ball method.

Experimental Procedure

The raw materials La_2O_3, and ZrO_2 were weighed to an accuracy of 0.001 grams and mixed in an agate jar for about 1 hour by human hand.and Raw materials were weighed to an accuracy of 0.01 gms and wet ball milled in an agate media for 12 hours, the mixed slurry was dried at 150 ^{0}C for 12 hours. The dried powder was mixed with 7.5 % Poly Vinyl alcohol (PVA) and compacted into circular pellets as shown in fig2

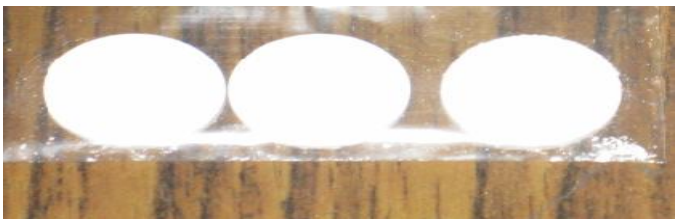


Fig 2, Circular pellets

The pellets were placed on an Alumina plate and were kept in Lenten Furnace., In this method Lanthanum Zirconate($La_2Zr_2O_7$) Powder was prepared from reaction between La_2O_3,(99%) with ZrO_2 (99%) at 900^0 c 6hrs.and1400^0 c for 6hrs soaking for 4hrs. Calcinations cycle is schematically in Figure3.

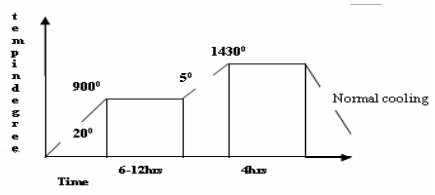


Fig 3: Calcination cycle

The calcined pellets were crushed in an Agate jar obtain very fine particles. The particles were sieved in 200 mesh sieve to obtain particle sizes of less than 53 microns. The crushed powder is ready for XRD analysis. The X-ray diffraction measurements were carried out on powdered samples using a high-resolution SIEMENS D 500 diffractometer. Filtered CuKα λ = 1.54056 Å (40 kV, 25 mA) was used. The diffraction diagram was measured from 20° to 60° in 2θ range with step size 0.02° (2θ) and 2 s counting time.

3.0.Results and discussions

Introduction

The results and discussions of characterization of lanthanum zirconate powders prepared from two methods such as

1.Agate jar method, 2.Wet ball mill method are presented in this paper.

3.1.Agate jar method

The XRD pattern of lanthanum based zirconia prepared by agate jar mixing is as shown

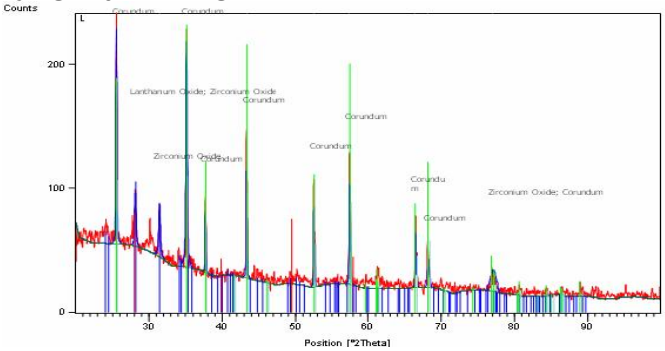


Fig 4: XRD pattern of lanthanum oxide based zirconia by agate jar method

Comparing the peak list obtained from XRD with the peaks of the standard ICDD values, most of the peaks with their respective intensities are not match so we can conclude that lanthanum zirconate was not formed. As there was no formation of zirconates, there was no scope for future work.

4.2. Wet ball mill method

In the Wet ball mill method grinded powders were placed in XRD Machine and the powdes are analysed at 0.2for 12hrs and after the calcinated pallets XRD analysis showed the presence of lanthanum hydroxide in place of lanthanum oxide. This is because of the absorption of moisture from the atmosphere by lanthanum oxide.The XRD pattern obtained for lanthanum oxide based zirconia is as shown in the figure5.

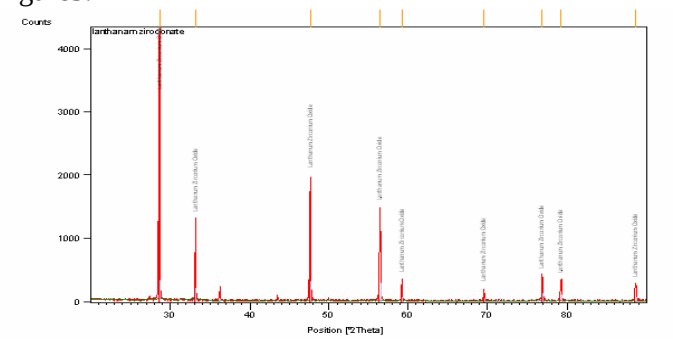


Fig5: XRD pattern of Lanthanum oxide based Zirconia

Pos[^{0}2θ]	Height[cts]	FwHm[2θ]	d-spac[A]	I[%]
28.6264/ 28.571	432095	0.1020	3.11581/ 3.12173	100.00/ 100.0
33.1693/ 33.109	127334	0.1020	2.69872/ 2.70350	29.47/ 26.2

Comparing the peak list obtained from XRD with the peaks of the standard ICDD values, most of the peaks with their respective intensities match. This confirms the formation of lanthanum zirconate.[5]

4.0. CONCLUSION

From the experimental results the following conclusions are drawn.,By agate jar method, there is no formation of zirconate phase due to higher particle size. In the wet ball milling since the particles are very fine (i.e. 1 to 5 microns) and also it has very fine grain size .Therefore it has very good affinity to undergo phase transformation when calcined at temperature 1435^{0}C.

5.0.REFERENCES

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