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Lanthanum Phosphate Based Ceramic Composite Coatings for High Emissivity Applications

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ABSTRACT

Lanthanum phosphate, a soft ceramic material, with low thermal conductivity, high melting point and high temperature phase stability is a good candidate for getting a tough ceramic coating for use at high temperatures. In this work, a novel high emissivity ceramic coating based on lanthanum phosphate ceramic pigment was developed. Initially, a black pigment based on lanthanum phosphate with an L* value of 34.71 was obtained through solid state reaction by suitable pigment addition and subsequent high temperature firing. This pigment was then brush painted on to alumina and stainless steel substrates using aluminium phosphate and sodium silicate inorganic binders and fired at suitable temperatures for setting of coatings. The average spectral emissivities of the coatings in the wavelength range 0.7μ m to 2.5μ m was above 0.9. The emissivity variation with porosity of substrate, temperature, roughness of coating and viscosity of binders was studied for getting an optimised coating with high emissivity. **Keywords:** Lanthanum Phosphate, Thermal Conductivity, Aluminium Phosphate, Orthophosphoric Acid, LaPO₄, NH₃, Aluminum and Alumina

I. INTRODUCTION

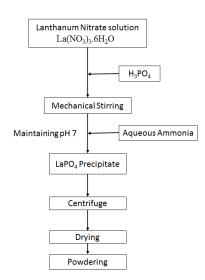
A ceramic is an inorganic, nonmetallic solid material comprising metal and a nonmetal atoms held by ionic or covalent bonds [1]. They can be mainly classified into amorphous, semi crystalline and crystalline ceramics. High melting temperature, high hardness, poor conductivity, high moduli of elasticity, chemical resistance and low ductility are some of the common properties of ceramics with known exceptions to each of these rules (e.g. piezoelectric ceramics, glass transition temperature, superconductive ceramics, etc.). The word ceramics is derived from greek word keramikos meaning pottery and the earliest ceramics made by humans were pottery objects made from clay, either by itself or mixed with other materials like silica, hardened, sintered, in fire [2]. Later these ceramics were glazed and fired to create smooth andcolored surfaces, decreasing porosity through the use of glassy, amorphous ceramic coatings on top of the crystalline ceramic substrates. Ceramics fiinds a number of applications in domestic, industrial and building products, as well as in a wide range of ceramic art. New

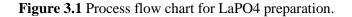
ceramic materials were developed for use in advanced ceramic engineering, such as in semiconductors in the 20^{th} century.

II. METHODS AND MATERIAL

Lanthanum phosphate is obtained by sol-gel method by the addition of orthophosphoric acid (H_3PO_4 , 85%, Merck Specialties Private Limited, India) to lanthanum nitrate hexahydrate salt (La(NO_3)_3.6H₂O, 99.99%, Alfa Aesar). A solution of La (NO_3)_3.6H₂O is made in distilled water. The amount of La (NO_3)_3.6H₂O and distilled water required for the solution is calculated on the basis of the amount of LaPO₄ required. The solution is stirred thoroughly using a mechanical stirrer for 30 minutes.

Orthophosphoric acid solution in distilled water is added drop by drop to the stirring solution. The solution is allowed to stir for about 30 minutes after the complete addition of orthophosphoric acid. The pH of the solution is checked using litmus paper. Now add a small amount of NH_3 drop by drop to convert the pH to 7. This conversion from acidic pH to basic aids the precipitation of LaPO₄ and allowed to precipitate completely. The LaPO₄ precipitate is filtered thoroughly using filter paper and Buckner funnel. The precipitate thus obtained is washed 5 to 10 times thoroughly with hot distilled water to remove the NH₃ content. The obtained precipitate is dried in a water bath arrangement. The dried LaPO₄ flakes obtained are crushed into powder using mortar and pestle. The process chart for LaPO4 preparation is given in figure 3.1.





III. Synthesis of LaPO4 Based Pigments And Compacts

Solid State Reaction method as dicussed below was used for pigment and compact preparation. For pigment and compacts production initially lanthanum phosphate prepared from the above route discussed was taken and mixed with powders available in the laboratory such as NiO, Co₂O₃, Co₃O₄, MnO₂, CuO etc. with a specific weight ratio. This composite mixture was then taken for wet ball milling using Zirconia balls having a weight of 3g each. The weight of powder to balls was chosen to be 1:3 for proper milling. Water-Propan-2-ol mixture with 1:1 volume ratio was taken as the ball milling medium. Ball milling was done in a 350ml milling plastic bottle. For 15g of powder mixture about 250 ml of (waterpropan-2-ol) was taken. It was then ball milled for 24 hours at an rpm of 300. Such long time of ball milling allows for proper and uniform mixing and size reduction of powders. The resulting mixture was then heated in a hot plate till all water and propan-2-ol gets evaporated. For NIR reflective pigments this precursor powder was heat treated at 12000C in a high temperature furnace. For obtaining black pigments and black ceramic compacts, the precursor powder was calcined at 6000Cfor 2 hours in an alumina crucible. For obtaining black pigments, calcined powders was heat treated at 12000C

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for 2 hours in an alumina crucible and for obtaining ceramic compacts, the calcined powders were compacted in a press under a pressure of 1 Ton with a holding time of 1 minute and then sintered in air atmosphere at 14000C for 1 hour in a high temperature furnace. Figures of some of the equiments and it's make used in pigment and compact production are given in fig 3.1



Ball Mill Unit

Fig 3.2 Hydraulic Press (make:Carver)

3.1 Synthesis of Binders And Coatings

Since organic binders do not have high temperature stability, inorganic binders where chosen. Coatings were prepared with two different inorganic binders.One was an alkali metal silicate binder (Sodium-Silicate) with SiO₂:Na₂O in the ratio 2:1. They are used for making heat resistant paints. Other was an acidic aluminium phosphate binder [38]. Acid aluminum phosphate refers to a liquid solution of phosphoric acid (H₃PO₄) and an aluminum salt (e.g., aluminum hydroxide), such that the proportion of acid is above that needed to form solid aluminum phosphate (e.g., Al(PO₃)₃, which is aluminum AlPO₄, which metaphosphate, and is aluminum orthophosphate). Hence the P/Al molar ratio in acid aluminum phosphate is typically much higher than 3. Elements other than aluminum (e.g., calcium) can be used in acid phosphates, but aluminum is most commonly used, due to the wide availability of aluminum salts and the importance of aluminum and alumina (Al2O3) among engineering materials. Phosphoric acid itself can be used for the binding and surface modification of materials (e.g., ceramics), but the addition of aluminum significantly enhances the bonding ability. Heat treatment is needed subsequent to application of the acid aluminum phosphate in binding or coating.

The heat treatment is partly for the purpose of drying and results in the formation of crystalline and amorphous aluminum phosphate solid phases, depending on the heating temperature and the possible interaction or reaction between the acid phosphate and the material to be bound or coated. Aluminum metaphosphate is a crystalline phase that is commonly formed after heating at 500–800°C.

For coatings, a pigment to binder weight ratio of 1:1.3 was chosen. Usually applications such as for spray painting, brush painting etc, the pigment to binder weight ratio is varied between 1:1 to 1:1.5. A smaller ratio below this region leads

to increased viscosity and painting or brushing may be very difficult, while a higher ratio above this region may degrade or affect the advantageous properties of the ceramic powders added. Hence an optimum ratio of 1.:1.3 within the allowed variation was chosen. As already discussed, every high emissivity coating will comprise of a refractory pigment, high emissivity agents and a suitable binder. With this regard, from the pigments synthesized from the above step, a black pigment was taken and coated with two different binders on to alumina and stainless steel 304 substrates. Samples coated with sodium-silicate binder was dried at 200°C and samples coated with aluminium phosphate was dried at 500°C in a low temperature furnace for 2 hours.

IV. RESULTS AND DISCUSSION

In this part, the results of the experiments conducted and discussions on that results based on the observations are done. The results obtained for pigments, binders, coatings and ceramic compacts in that order is studied and discussed. NIR reflective pigments for cool roof appication and black pigments for high emissivity applications were produced and characterized separately as discussed below.

4.1 Near Infrared Reflecting Pigments

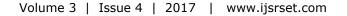
NIR curves for the pigments is given in Fig 4.2 which shows that lanthanum phosphate is an excellent candidate for these kind of applications. By small amount of pigment addition and firing, colours such as grey and green was obtained. The composition for their production and the codes for the samples are given in table 4.1.

Table 4.1 Composition and code for pigments

| LaPO ₄ | CuO | MnO ₂ | Co ₂ O ₃ | NiO | Code |
|-------------------|-----|------------------|--------------------------------|-----|----------|
| 95 | 2.5 | 2.5 | | | LCM |
| 93 | 2.5 | 2.5 | 2 | | LCMCo |
| 93 | 2.5 | 2.5 | | 2 | LCMNi |
| 100 | | | | | $LaPO_4$ |

Figure 4.1 shows the pigments which were obtained which shows Co^{3+} addition changed the colour from grey to green for lanthanum phosphate.



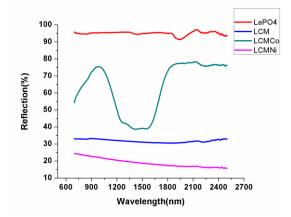


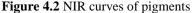


LCMNi



Figure 4.1 Pigments obtained





Avg: NIR reflectance for LaPO₄ was above 95% which shows that doping and obtaining coloured pigments based on lanthanum phosphate may yield coloured pigments with high NIR value. Similarly, lanthanum phosphate is an excellent refractory material and has low thermal conductivity at elevated temperature(discussed earlier). The properties such as low thermal conductivity, non-reactivity with acids and bases, non-wettability along with NIR reflection shows that lanthanum phosphate can be an ideal candidate for heat shielding purposes such as for thermal barrier coatings. As we know, at high temperatures the majority of the thermal radiations emanating from the source will be of shorter wavelengths(NIR) and as shown in fig 4.2 lanthanum phosphate is an excellent NIR reflector and hence it could be an excellent heat shield material also. The solar NIR reflectance (R*) for the pigments calculated on the basis of equation1 is given in table 4.2. For LCMCo coded green pigment, the calculated solar NIR reflectance was about 62%. Similarly as we see from the solar spectral irradiance data

(figure 1.6), most of the energy of the solar radiation in the NIR part is concentrated in the region from 700 to 1100 nm. As we see from the NIR curve of LCMCo green pigment, the reflectance in the above said region is high, hence it will be an ideal cool pigment. The other two pigments were grey in colour with LCM pigment having an R* value of 32%. R* values of some commercial pigments and of some pigments taken from literature with their colour in bracket is given in table 4.3.

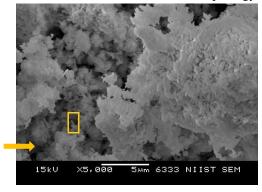
| Table 4.2 Solar NIR | reflectance | (R^*) |) of pigments |
|---------------------|-------------|---------|---------------|
|---------------------|-------------|---------|---------------|

| Pigments | Solar NIR Reflectance,R* (%) |
|---------------------------|------------------------------------|
| LCM (grey) | 32 |
| LCMCo (green) | 62 |
| LCMNi (grey) | 21 |
| LaPO ₄ (white) | 95 |

 Table 4.3 NIR Solar reflectance of some pigments from literature

| Pigments | NIR Solar Reflectance (%) |
|--|---------------------------------|
| White TiO ₂ - rutile | 87 |
| Green (Y ₂ BaCuO ₅) | 50 |
| Commercial green (Cr ₂ O ₃) | 55 |
| Blue(La-Sr- Cu-Silicate) | 67 |
| Red(Tb-doped- Y-cerate) | 80 |

Fig 4.3 shows the SEM of lanthanum phosphate (fig 4.3 a) and the green pigment LCMCo (fig 4.3 b) developed. Lanthanum phosphate powder had a rod like morphology, highlighted in fig 4.3 a(consistent with the literature reports) but showed some amount of agglomeration possibly because the powders were not sonicated prior to it's deposition in the carbon tape. However, the green pigment did not show any agglomeration and it showed a sheet like morphology.



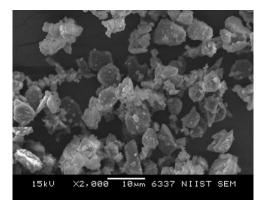
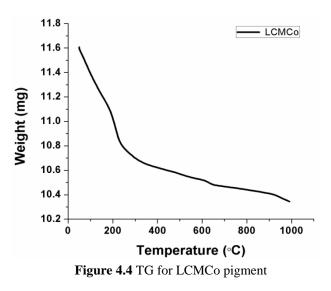


Figure 4.3 (a) SEM of LaPO₄Fig 4.3(b) SEM of green pigment

Fig 4.4 gives the TG of the as prepared LCMCo pigment. The total calculated weight loss upto 1000^oC for the pigment was 10.88%. Interestingly, lanthanum phosphate had a slightly higher weight loss of 11.11%. The TG curve of the lanthanum phosphate was similar to that of LCMCo as in figure 4.4.



From figure 4.4 we can see that the slope of the curve is greater for a temperature from about 30°C to 300°C. Within this region curves with two different slopes, first one between 30° C to 180° C and second one between 180° C to 300° C can be seen. The initial slope from 30°C to 180°C is a steep one and is due to the loss of absorbed water during ball milling. The slope in the region from 180° C to 230° C is the highest because of the combined effect of loss of water due to absorption and adsorption. This curve has a maxima at 220°C which is because of the loss of water of crystallisation associated with lanthanum phosphate. Apart from the weight loss due to loss of water, no major reactions are happening till 1000° C for 5^oC heating rate. However as it can be seen, since the heating rate was 5° C their was a slight weight loss even till 1000°C, but it was negligible compared to the initial weight loss. Considering these factors, for the same heating rate, a heat treatment temperature of 1200°C and a holding time of one hour was chosen for the pigment production.

Figure 4.5 gives the particle size of the pigments.As we can see, the particle sizes of all the pigments produced was higher than that of the as prepared lanthanum phosphate powder. This is because of the heat treatment process adopted for their production($1200^{\circ}C$ 1 hr). The average particle size of the as prepared lanthanum phosphate by the aqueous precipitation method was about 267 nm consistent with the literature results (265 nm).

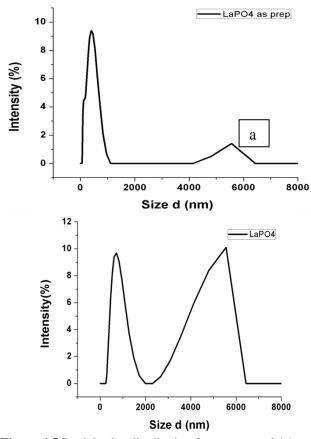


Figure 4.5 Particle size distribution for as prepared (a) and heat treated (b) LaPO₄

Both as prepared and heat treated lanthanum phosphate powders showed bi- modal distribution of particles However, as can be seen from fig 4.5 this bi-modal distribution is not as pronounced for the as prepared powder compared to the heat treated lanthanum phosphate powder. The average particle size increase from 267 nm from as prepared powder to 1.09 μ m for 1200⁰C 1 hr heat treated lanthanum phosphate powder. Similarily, there was also an increase in particle size of the coloured pigments. The particle size of the green LCMCo pigment was about 1.86 μ m and that of LCMNi grey pigment was 2.14 μ m (fig 4.6). Both showed an uni-modal distribution of particle size and their particle sizes were above that of heat treated LaPO₄ showing that pigment addition leads to increase in particle size as well.

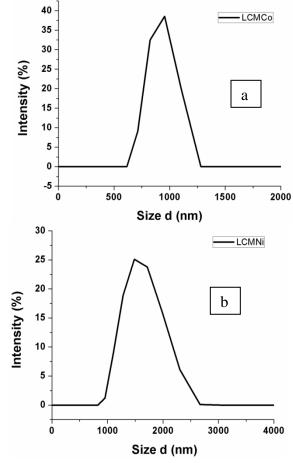


Figure 4.6Particle size distribution for the produced green (a) and grey (b) pigments

A lower particle size causes better NIR reflection. The XRD patterns of the heat treated lanthanum phosphate, green (LCMCo) and grey (LCMNi) pigments plotted together are given in figure 4.7. From figure 4.7 (a) it can be seen that the peaks of both heat treated lanthanum phosphate and LCMNi coincides indicating that LCMNi pigment is just a composite pigment. However for for LCMCo green pigment, the peaks donot coincide as seen in figure 4.7 (b) which indicates that a new material with a different structure has been formed.

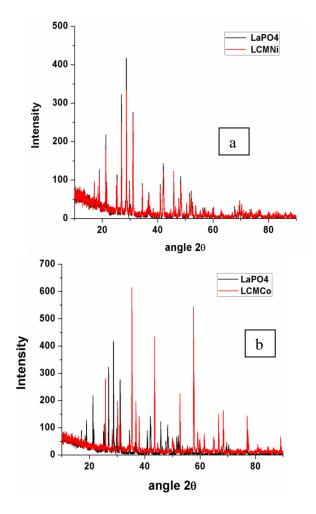


Figure 4.7 XRD of grey LCMNi and green LCMCo plotted together with LaPO₄ produced under identical conditions.

4.2 SYNTHESIS OF BLACK PIGMENT'S & IT'S CHARACTERISATION FOR HIGH EMISSIVITY COATINGS

Black colour is a good absorber, particularly in UV-Visible range and black coloured pigment/s based coatings are used for getting solar-selective surfaces with high absorbance in UV-Vis region and low IR emittance. A low emissivity, in many cases is achieved by controlling the thickness of the coatings. Normally, emissivity increases with increase in thickness of the coating and there is a critical thickness of coating upto which emissivity increases after which it remains constant. Hence the initial objective was to prepare a black pigment based on lanthanum phosphate by pigment addition and then to obtain a coating with that black pigment for high emissivity applications.

Initially a series of experiments were done by varying the compositions, weight percentages of the components for obtaining a black pigment. The details of which are given in table 4.4. As already discussed, the main characterization for the black pigment is it's L* value and one with a low L* value was chosen for coating.

Table 4.4 Composition, weight percentages and L* values of the pigments.

| Pigment code | LaPO ₄ | CuO | Co ₂ O ₃ | MnO ₂ | Co ₃ O ₄ | NiO | Cu-slag | L* |
|-----------------|-------------------|-----|--------------------------------|------------------|--------------------------------|-----|---------|-------|
| S1 | 95 | | 1 | 2 | | 2 | | 50.82 |
| S2 | 95 | | 2 | 2 | | 1 | | 46.76 |
| S3 | 95 | | 2 | 1 | | 2 | | 53.02 |
| S4 | 90 | | 4 | 4 | | 2 | | 42.93 |
| S5 | 85 | | 6 | 6 | | 3 | | 42.04 |
| S6 | 80 | | 8 | 8 | | 4 | | 42.61 |
| S7 | 90 | 4 | | 4 | | 2 | | 41.07 |
| S8 | 80 | 8 | | 8 | | 4 | | 40.11 |
| S9 | 70 | 12 | | 12 | | 6 | | 39.61 |
| S10 | 75 | | | 8 | 9 | 8 | | 34.71 |
| S11 | 85 | 4 | | 4 | | 2 | 5 | 45.31 |
| S12 | 80 | 4 | | 4 | | 2 | 10 | 46.11 |
| S13 | 75 | 4 | | 4 | | 2 | 15 | 49.17 |

The TG and DTA curves for the as prepared black pigment (S10) is given in figure 4.8. The heating rate was set to be 5ºC/min, identical to the actual processing condition. As we can see the total weight loss is about 18 %. The major weight loss is due to the loss of water which is happening throughout the process of heating. Since the heating rate is quite high $(5^{\circ}C/min)$, the weight loss happens even at higher temperatures and is negligible towards higher temperatures. Similarly, the weight of the powder before and after heat treatment was noted using a weighing machine. Interestingly, the weight loss of the as prepared powder after high temperature firing at 1200° C for 2 hours (taken manually) was only about 13% (average of 3 readings). From the differential of the TG curve plotted in the right axis of figure 4.8 a, the DTG curve shows a peak at 220° C where the weight loss is maximum and is due to an endothermic reaction at 220^oC as shown in DTA (figure 4.8 b) of the pigment. The endothermic reaction is due to the loss of water of crystallization associated with lanthanum phosphate.

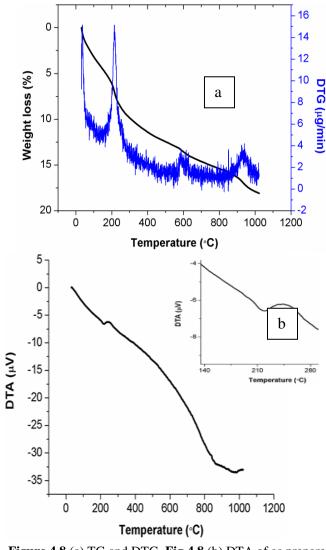


Figure 4.8 (a) TG and DTG, Fig 4.8 (b) DTA of as prepared black pigment

Figures of the as prepared and heat treated powder is given in figure 4.9. The UV-Visible reflectance and the L* values of the as prepared and 1200° C heat treated powder is given in fig 4.10. The heat treated powder showed low absorbance and L* values possibly due to the loss of water due to heat treatment.



Fig 4.9 (a)as prepared and (**b**) heat treated black pigments. Heat treatment was done due to many reasons. The primary reason is that, eventhough the water and propan-2-ol mixture milling medium evaporates after the heating using hot plate, there is considerable amount of water content left in the as prepared powder and this is clear from the TG of the as prepared powder as shown in fig 4.8 (a). If we prepare the paint slurry with the as prepared black pigment, fire & hold it at elevated temperature $(300^{\circ}C-500^{\circ}C)$ for setting of coating, it may not yield good result as the residual water in the as prepared black pigment starts evaporating and leads to disintegration and poor properties of the coating affecting the adhesion strength of the coating with the substrate. Secondly, the particle size increases with heat treatment, which is advantageous in this case (discussed later). Thirdly, high temperature treatment may facilitate formation of phases such as spinels which help in increasing emissivity as discussed earlier.

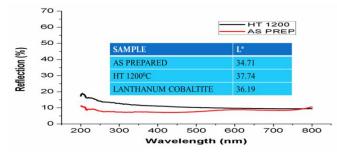
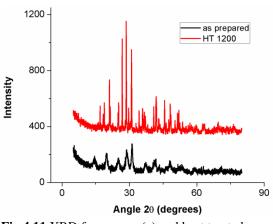
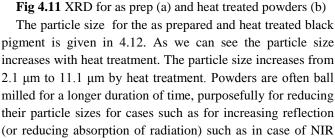


Fig 4.10 UV-Visible reflectance and L* values of as prepared and heat treated black pigments

L* value of lanthanum cobaltite black pigment prepared from lanthanum-oxide and cobalt-oxide from literature [39] is also given in figure for comparison. The XRD data for the as prepared and heat treated powder is given in figure 4.11 a and b respectively. As we can see the powder becomes well crystallined due to heat treatment and is indicated by the sharp peaks of the heat treated powder compared to as prepared powder. The major peaks of the as prepared powder corresponded to that of lanthanum phosphate. Similarily, the major peaks of 1200^oC 2 hour heat treated powder correspond to lanthanum phosphate. Some amount of spinel phases such as MnCo₂O₄, CoCo₂O₄and other phases such as lanthanum cobalt oxide phosphide was also formed in case of heat treated powder.





reflecting powders. As already discussed, the ball milling time was 24 hours. Eventhough, such prolonged time for milling allows uniform mixing of powders, the particle sizes of the powders reduces significantly which can be seen from figure 4.12 (a) where the majority of particles acquire smaller particle size. Since, the aim was to increase absorption and hence emissivity, a heat treatment was done to achieve a powder with greater particle size and this powder was used for coating.

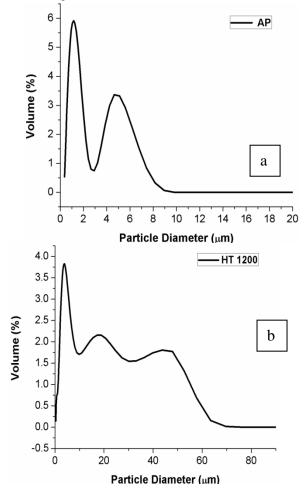


Figure 4.12 Particle size curves for the as prepared (a) and heat treated (b) powders.

The SEM of the as prepared powder is given in figure 4.13. SEM agrees with particle size data (fig 4.12 (a))and shows particles with two morphologies and sizes as shown in figure 4.13 (c). Lanthanum phosphate is a soft material and the prolonged ball milling can decrease it's size. Therefore, the smaller particles could be lanthanum phosphate and the bigger rod like particles could be any one of the transition metal-oxides. Particles with dual morphology and sizes (of 1.9 μ m and 5.3 μ m) can be seen in the SEM (fig 4.13 (c)). Figure 4.13 (b) shows that some amount of agglomeration has happened with lanthanum phosphate particles. The rod morphology particles (figure 4.13 (a)) shows a leaf like or feather like morphology when it is zoomed (fig 4.13 (c) and fig 4.13 (d)).

The heat treated black pigment was used for making coatings.

4.3 SOLAR ABSORPTION AND SPECTRAL EMISSIVITIES OF COATINGS & COMPACTS

The coatings were brush painted on alumina discs and SS 304 substrates. Alumina powder was compacted and fired for making alumina with diameter 40 mm and thickness 5 mm (fig 4.14). The calculated Archimedes density of the alumina disc was 2.51g/cc. Figure 4.15 shows stainless steel coated using sodium silicate binder.

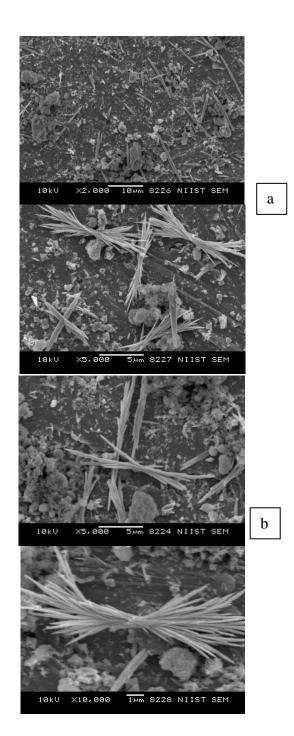


Figure 4.13 SEM of black pigment at different magnifications.

The UV-Visible reflectance and calculated solar absorptance for stainless steel coatings named SS1,SS2 so on are given in figure 4.16 and table 4.5 respectively. As we can see bare SS substrate has a higher reflection and hence low absorption. But the coating reduced the reflection.All the coatings showed similar reflectance curves. A maximum solar absorption of 0.95 was achieved. Such high solar absorbance coating may be used for harvesting solar energy for purposes such as electricity production, heating etc.Normal solar absorber coatings use organic binders which are not high temperature stable and hence the efficiency of power production is low. However, use of inorganic binders such as the one discussed above solves this problem and higher efficiency may be achieved by using the coatings at higher temperatures.



Figure 4.14 alumina compacted and fired

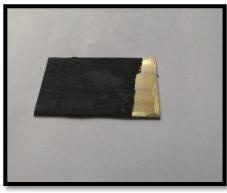


Figure 4.15 Coated stainless steel

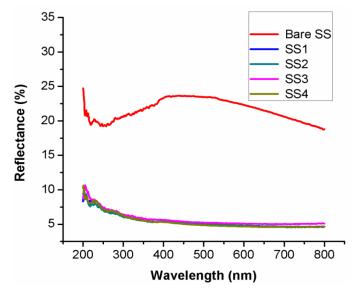


Figure 4.16 UV-Vis reflectance of coated SS samples.

| SAMPLE | $\begin{array}{c} \text{SOLAR} \\ \text{ABSORPTANCE} \\ (\alpha_s) \end{array}$ |
|--------|---|
| SS1 | 0.95 |
| SS2 | 0.94 |
| SS3 | 0.94 |
| SS4 | 0.95 |

 Table 4.5 Solar absorptance of coatings.

The spectral emissivity of coatings in the wavelength range 0.7 to 2.5 µm is given in figure 4.17 and average spectral emissivities for samples are listed in table 4.6. As we can see, the spectral emissivity rises towards the shorter wavengths and the curves can be mainly divided into three regions. First region is from 2.5 µm to 1.75 µm where the coatings exhibit emissivity below 0.9. The second region is from 1.75 µm to 0.875 µm where the coatings exhibit emissivity just above 0.9. The third region is from 0.875 to 0.7 µmwhere the coatings exhibited emissivity well above 0.9 (0.93-0.94). Higher emissivities at shorter wavelength's correspond to a higher amount of thermal radiation at higher temperatures by Wein's displacement law of thermal radiation. The ideal radiant emitter for for use with GaSb photovoltaic cells for power (electricity) production would have an emissive power spectrum with maximum radiation power between about 1.0 and 1.7 µm. With this regard, the coatings developed had shown emissivity values above 0.9 in the wavelength range 1.0 to 1.7 μ m so that these may be used for applications such as one discussed above. Coatings with emissivity values greater than 0.9 are called high emissivity coatings.

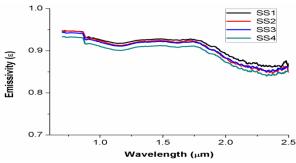


Figure 4.17 Spectral emissivity of coated SS samples

Table 4.6 average spectral emissivities od coated SS samples

| SAMPLE | SS1 | SS2 | SS3 | SS4 |
|--|------|------|------|------|
| AVG:SPECTRAL EMISSIVITY (ε_{λ}) | 0.91 | 0.90 | 0.90 | 0.88 |

Coatings on alumina using aluminium phosphate and sodium-silicate binder is given in figures 4.18 and 4.19 respectively.



Figure 4.18 Coatings on alumina using aluminium phosphate binder.



Figure 4.19 Coatings on alumina using sodium silicate binder

The absorptance and emissivities of coatings on alumina are given in figure 4.20. Here NS denotes coatings prepared with sodium silicate and AP denotes coatings prepared with Aluminium Phosphate binder. There was an increase of about 5% in emissivity of the samples coated with sodium silicate binder. This will be discussed later. The calculated solar absorptance of all the samples are 0.9 and above which indicates that these may be good candidates for volumetric solar receivers. Normal candidates for these kind of

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applications are SiC,Al₂O₃ etc. SiC suffers from oxidation and high emissivity problems, while Al2O3 has a low solar absorptance due to it's inherent white colour as shown in figure. However the coated samples showed excellent solar absorption. Solar absorptance and average spectral emissivities of coated alumina samples are given in table 4.7.

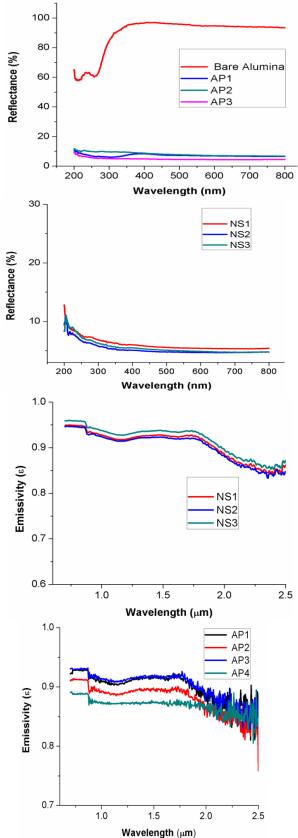


Figure 4.20 UV-Vis reflectance and spectral emissivities of coatings on alumina.

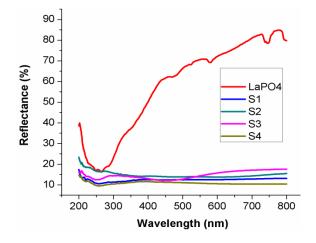
Table 4.7 Solar absorptance and average spectral emissivity of coatings on alumina.

| SAMPLE | AP1 | AP2 | AP3 | AP4 | NS1 | NS2 | NS3 |
|------------------------------|------|------|------|------|------|------|------|
| α, | 0.92 | 0.90 | 0.93 | 0.90 | 0.94 | 0.95 | 0.95 |
| AVG: ε_{λ} | 0.90 | 0.88 | 0.90 | 0.87 | 0.93 | 0.91 | 0.93 |

 Table 4.8 Solar absorptance and spectral emissivity of sintered compacts.

| SAMPLES | CO | COMPOSITION IN WEIGHT % | | | | | |
|------------|----------|--------------------------------|-----|-----|---------|------|------|
| | $LaPO_4$ | Co_3O_4 | CuO | NiO | MnO_2 | | |
| S 1 | 94 | 2 | 2 | 2 | 0 | 0.88 | 0.85 |
| S2 | 94 | 2 | 2 | 0 | 2 | 0.85 | 0.81 |
| S3 | 94 | 2 | 0 | 2 | 2 | 0.85 | 0.80 |
| S4 | 94 | 0 | 2 | 2 | 2 | 0.90 | 0.85 |

Table 4.8 gives the composition, solar absorptance and average spectral emissivity of sintered ceramic compacts. The calculated solar absorption of lanthanum phosphate ceramic was 0.42. Figure 4.21 gives the UV-Vis reflectance and spectral emissivity of compacts. The spectral emissivity curves are below that of spectral emissivity curves for coatings. Similarily, the compact S4 shows some amount of spectral selectivity. The emissivity goes for a decreasing trend towards the mid-infrared wavelength, while the emissivity increases for shorter wavelength's indicating that it might emit significantly at higher temperatures and at lower & mid-temperatures it might not emit that much. This kind of spectrally selective ceramic emitters may be used for applications such as Thermo Photo Voltaic Radiators.



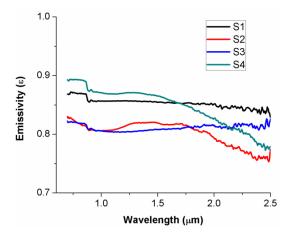


Figure 4.21 UV-Vis reflectance and spectral emissivity of sintered ceramic compacts



Figure 4.22 Sintered Ceramic Compacts.

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V. CONCLUSION

Novel energy saving materials and coatings based on lanthanum phosphate has been developed and tested. Lanthanum phosphate with it's excellent near infrared reflectance (95.44%) could be used for making heat shield applications such as thermal barrier coating. Similarily, it was shown that lanthanum phosphate could be used for making coloured pigments with high NIR reflectance values for cool roof applications. A green pigment with NIR reflectance of 67% was developed. The black pigment obtained via solid state reaction method showed L* value of 34.71. The coatings obtained with black pigment showed high solar absorbance and high infrared emissivity (both above 0.9). Dark sintered ceramics of lanthanum phosphate obtained through pigment addition, compaction and firing showed some amount of spectral selectivity with a good solar absorption of 0.9 and thermal emissivity of 0.85. The factors affecting emissivity was identified and studied. Based on the studies, it can be concluded that **rough ceramic coating, with a controlled thickness on a porous substrate using a viscous binder could be an excellent emitter**.

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