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Plasma spray-deposited lanthanum phosphate coatings for protection against molten uranium corrosion

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ABSTRACT

Lanthanum phosphate is recommended as an effective barrier against molten uranium and its alloys. $LaPO_4$ has a high melting point, is inert towards reactive metals and has low thermal conductivity, which make it an ideal candidate for corrosion barrier coatings in molten metal environment. Studies on plasma deposition of lanthanum phosphate, its characterization and interaction with molten uranium are reported in this paper. Lanthanum phosphate was synthesized by chemical route and the as-synthesized powder was converted to free flowing powder for plasma spray deposition. Lanthanum phosphate coatings were prepared on stainless steel substrates and the deposits were characterized for phase composition using X-ray diffraction (XRD), Raman spectroscopy and Fourier transformed infrared spectroscopy (FTIR). Results showed that the coatings retained the monazite structure of $LaPO_4$ precursor powder. Microstructure of the coatings and interface by scanning electron microscope (SEM) revealed good bonding between the phosphate layer and substrate. Porosity of the coatings was observed to decrease with the increase in deposition power from 10 kW to 20 kW. Adhesion strength of the coatings was found to increase with deposition power. Corrosion studies of coated $LaPO_4$ in molten uranium showed that lanthanum phosphate does not chemically react with molten uranium and $LaPO_4$ coating offers adequate resistance to the substrate against corrosion attack by molten uranium.

1. Introduction

Lanthanum phosphate monazite has a high melting point and does not react with molten material including uranium and its alloys. Lanthanum phosphate coatings are recommended for use in many high temperature applications to protect critical components from corrosive wear by reactive molten metals such as uranium. $LaPO_4$ deposited by atmospheric plasma spray technique has been suggested for corrosion prevention application of crucibles and other components that handle molten metals and alloys [1–3].

Atmospheric plasma spray (APS) technique is widely used for depositing ceramics for corrosion barrier and thermal barrier coating (TBC) applications [4–9]. Plasma sprayed coating provides protection against corrosion and increases the life of the components. In plasma spraying, ceramic or refractory powder is injected into the thermal plasma jet, where it undergoes rapid melting and gets accelerated. These molten droplets propelled at high velocities, typically exceeding 100 m/s, impact on the substrate surface to form adherent coating [10, 11].

Anhydrous lanthanum phosphate, $LaPO_4$ (monazite) has monoclinic structure and is stable up to its melting point [2]. By virtue of its high melting point (2070 °C) [12], high thermal expansion co-efficient, excellent phase stability and low thermal conductivity (1.3 W/mK at 1000 °C) [13], it is recommended for high temperature thermal and corrosion barrier applications [1]. Table 1 lists some of the thermo-physical properties of lanthanum phosphate [12–17].

Plasma spray deposition of $LaPO_4$ on stainless steel and graphite substrates was reported by Ananthapadmanmabhan et al. [2]. The authors reported that $LaPO_4$ did not react with molten uranium. Seung-Ho Kim et al. [3] successfully prepared lanthanum phosphate (10–40%) with 3YSZ coatings on stainless steel substrates using atmospheric plasma spray (APS) method for TBC applications. The authors observed that the coatings consisted only of $LaPO_4$ and YSZ phases. Sucharita Sinha et al. [18] successfully coated monazite lanthanum phosphate using PLD technique.

The present paper reports plasma spray deposition of lanthanum phosphate and characterization of the coatings. Plasma spray deposition was carried out using the 40 kW atmospheric plasma spray system developed at the Laser & Plasma Technology Division, Bhabha Atomic Research Centre. The spray deposition was carried out on stainless steel substrates at plasma power levels of 10 kW to 20 kW. X-ray diffraction

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Table 1
Thermo-physical properties of $LaPO_4$.

Melting point Tm (°C)	2070
Thermal expansion co-efficient α (K^{-1})10^{-6}	9.6
Thermal conductivity at 1000 °C W(m-K)$^{-1}$	1.8
Vickers hardness of bulk sample (GPa)	4.86–5.5
Specific heat capacity (Cp) at 1600 °C JK^{-1} mol^{-1}	173

and Raman spectroscopy were used for characterizing the phase structure of the coatings. Scanning electron microscopy (SEM) was used for analyzing the micro-structural features. FTIR was used to characterize the P–O bond structure. Tensile adhesion testing was used for determination of coating adhesion. Corrosion studies and compatibility of lanthanum phosphate with molten uranium and corrosion protection of the underlying substrate by $LaPO_4$ coatings were followed by differential thermal analysis.

2. Materials and methods

2.1. Synthesis of plasma grade powder

The flow chart for preparing spray grade lanthanum phosphate powder is shown in Fig. 1. Lanthanum phosphate was prepared by chemical route using 99.99% pure lanthanum oxide powder and ortho-phosphoric acid (85%) as the starting materials. Lanthanum oxide and phosphoric acid were taken in the molar ratio of 1:2 and the reaction mixture was vigorously mixed using pestle and mortar. In order to ensure complete conversion of the oxide into phosphate, phosphoric acid was taken in excess. The reaction mixture was diluted with distilled water and the precipitate was allowed to settle. The precipitate was cleaned several times with distilled water to make it acid-free. The precipitate was then dried at 80 °C for 2 h in hot air oven. The diffraction pattern of the as-precipitated lanthanum phosphate after drying at 80 °C was seen to match with that of hydrated lanthanum phosphate ($LaPO_4$ 0.5H_2O), having rabdophane structure corresponding to JCPDS 46-1439 (Fig. 2). The agglomerated soft chunks of the precipitate, thus obtained, were then fired at 1600 °C for 4 h in a high temperature box furnace. The fired chunks were carefully crushed and sieved to get powder fraction in the range of 38–75 μm, 75–106 μm and 106–125 μm. Powder chunks of size >125 μm were again crushed, sieved and this process was continued to obtain the required quantity of plasma spray grade powder in different ranges of particle size. The spray grade powder thus prepared was used as the feedstock for plasma spray deposition. Fig. 2 shows the XRD pattern of the feedstock powder showing monazite structure corresponding to JCPDS card 84-0600.

2.2. Plasma spray deposition of lanthanum phosphate

Plasma spray deposition was carried out using 40 kW atmospheric plasma spray facility developed at the Laser & Plasma Technology Division. The torch consists of a thoriated-tungsten cathode (10 mm diameter) and a conical tipped copper anode nozzle, 7 mm in diameter. The electrodes are cooled by water and a Teflon insulator separates the electrodes. A mixture of argon and nitrogen was used as the plasma gas, which was injected through a side inlet in the insulator segment. Spray grade powder was fed into the plasma jet through an internal injector hole (2 mm diameter) on the anode nozzle located 3 mm before the exit of the nozzle. MEC PF 3600 powder feeder was used for feeding lanthanum phosphate powder into the plasma jet. Lanthanum phosphate powder of size 38–75 μm was used for plasma spraying experiments. The flow rate of the powder was maintained at about 10 g per minute

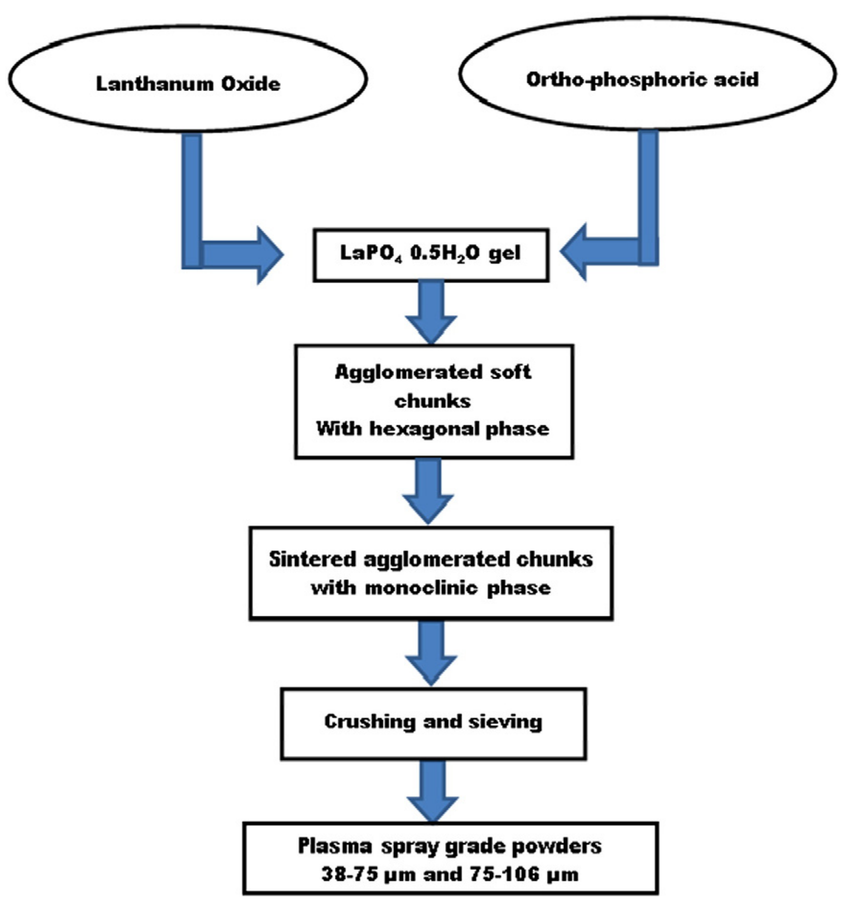

Fig. 1. Flowchart diagram for synthesis of plasma spray grade lanthanum phosphate powder production for plasma spray coatings.