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Processing window development for laser cladding of zirconium on zirconium alloy

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ABSTRACT

Zirconium is commonly used in nuclear, chemical processing and biomedical applications due to its low thermal neutron cross-section, relatively high corrosion resistance and great biocompatibility. In this study, powder spray laser additive manufacturing technology was used to deposit commercially pure zirconium on zirconium alloy substrates. The clad quality was assessed based on deposition rate, dilution, geometrical circularity and presence of defects. The effect of laser power, laser scan speed, laser spot size and powder feed rate on the clad quality was investigated. Defect-free clads, suitable for laser additive manufacturing were selected based on the analysis of process parameters. Zirconium clad could be deposited at a deposition rate of 0.80 g/min with a dilution rate of 50%.

1. Introduction

According to Lustman and Kerze (1955), low neutron absorption cross section and high corrosion resistance of zirconium and its alloys make it ideal for nuclear industry. In a study to evaluate the use of zirconium components for hip and knee implants, Balla et al. (2009) reported them as being hypoallergenic, biocompatible and osteoconductive. The researchers found that oxidized zirconium provided an excellent site for the growth of human osteoblast cells and resulted in higher wear resistance than commercially pure titanium and zirconium. AWS (2012) mentions that zirconium and its alloys also find use in chemical processing industry for their high corrosion resistance to most acids, strong alkalis, salt solution and molten salts.

Considering the high value and complex designs often involved in many of these nuclear, medical and chemical processing industry applications, the laser additive manufacturing of zirconium is an attractive option. However, the available literature on depositing zirconium on a zirconium based substrate is fairly limited.

Another potential application for zirconium involves the production of metallic glass. In a study to laser clad preplaced zirconium powder on an austenitic stainless steel substrate, Wu and Hong (2000) attributed the formation of metallic glass to the high glass forming ability of zirconium and the high cooling rates created by laser. The clad showed a duplex microstructure with regions of austenite and metallic glass, and exhibited a hardness that was 1.8–2.6 times higher than the substrate. Monfared et al. (2013) found that zirconium based metallic glasses showed great biocompatibility and potential for use as biomedical devices. Arora et al. (2013) found that zirconium based metallic glass showed higher hardness and cavitation erosion resistance than a commonly used hydro-turbine steel. It follows that laser additive manufacturing technology can be used to clad conventional parts with metallic glasses, with potential for creating 3D structures.

Although zirconium offers several highly desirable properties, it also has some shortcomings. NFPA (1996) emphasizes that zirconium fines and dust are highly combustible and care needs to be taken while handing them. Danielson and Sutherlin (2004) recommended using an active polishing method with a hydrofluoric acid solution to prepare zirconium samples for optical microscopy. This required careful metallography procedures and keen atten-

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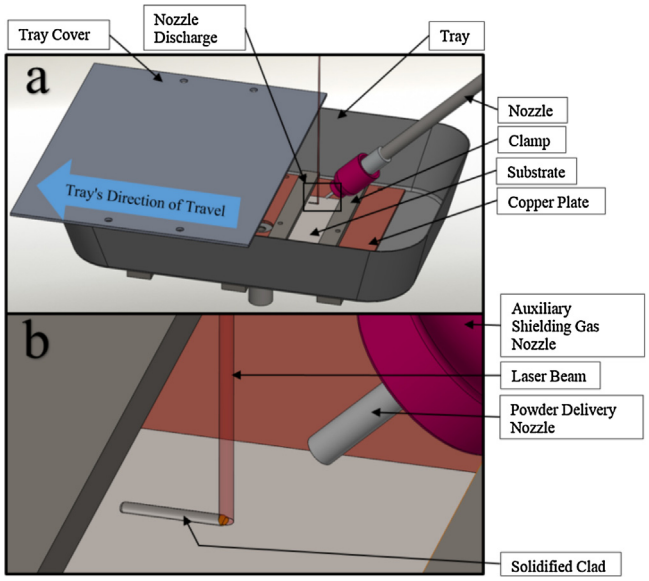

Fig. 1. Isometric schematic of the setup used during experiments; a: overall view; b: close up of the nozzle discharge.

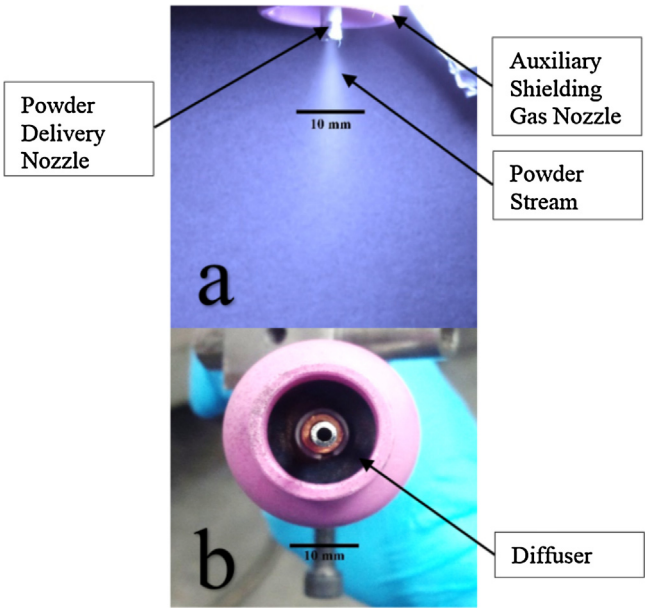

Fig. 2. Nozzle discharge; a: side view; b: bottom view.

tion to safety. Bláhová et al. (2009) reported that zirconium and its alloys react very readily with atmospheric gases at high temperatures. Additionally, AWS (2012) reported that zirconium welds were also susceptible to iron and carbon contamination. It follows that cladding of zirconium would require thorough cleaning of substrate prior to cladding and ample inert gas shielding during cladding.

To the best of authors' knowledge, little published research exists on depositing pure zirconium powder onto a zirconium alloy substrate. There has only been limited prior work on laser cladding of Zr powder on other alloys. Balla et al. (2009) and Baloyi et al. (2014) focused on depositing zirconium based coatings on titanium based alloys; Yue et al. (2012) deposited a pure zirconium coating on a AZ91D magnesium alloy; and Wu and Hong (2000) deposited zirconium onto an austenitic stainless steel substrate. None of these publications evaluate the use of zirconium or zirconium based alloys for laser additive manufacturing (i.e., by depositing onto zirconium based material), and do not study the role of deposition parameters on clad quality.

The present work represents a pioneering attempt to deposit pure zirconium on a zirconium alloy substrate without the use of an enclosed inert gas environment, but rather only using localized shielding. This is also significant in that most procedures involving high temperature processing or welding of zirconium recommend a controlled atmosphere; however, the nozzle design utilized here may offer sufficient shielding to avoid this requirement. In this light, the influence of changing process parameters on the resultant clad geometry was assessed using this more flexible system.

2. Experimental setup

Fig. 1 is an isometric schematic of the experimental setup used during the laser cladding experiments. The substrate was mounted onto a copper backing, within a specially designed tray to capture excess powder. The tray was mounted on a Computerized Numerical Controlled (CNC) machine that allowed controlled motion in the plane perpendicular to the laser beam. The energy provided by the laser created a melt pool on the substrate. A lateral nozzle was used to deliver powder into the melt pool. The increase in volume of melt pool created by the additional powder resulted in the clad.

A Sulzer Metco Twin-10-C gravity based powder feeder with metering wheel was employed. To send the powder through the powder delivery nozzle, an argon flow rate of 3.5 dl/min was used. A fiber laser (YLR-1000-IC by IPG Photonic) was used for the experiments, with a wavelength of 1070 nm. Motion was controlled by a Model 904-1 Fadal CNC machine.

Fig. 2 shows a close up of the discharge end of the nozzle. The nozzle was concentric; the inner nozzle delivered powder while the outer nozzle provided shielding. Spherical zirconium powder of 99.6% purity was used, which had a size range from 105 μm to 53 μm in diameter. Argon was used as the carrying gas for the inner nozzle. Argon was also used as the shielding gas in the auxiliary shielding nozzle. The purity of Argon gas was at least 99.995%. The outer nozzle was made using components of a Gas Tungsten Arc Welding (GTAW) torch. A diffuser at the discharge end of the outer nozzle ensured that the shielding gas flow was laminar. During cladding, a gas flow rate of 30CFH was maintained through the outer nozzle.

The angle of the nozzle to the substrate was $40 \pm 5^{\circ}$. The outer shielding gas nozzle had a discharge diameter of 15.9 mm. The inner powder delivery nozzle had a discharge diameter of 1.78 mm and extended 15.9 mm out of the outer nozzle. The inner powder delivery nozzle was aligned such that its tip was 11–12 mm from the substrate.

The substrate consisted of cold rolled sheets with dimension of 51 mm × 153 mm × 1.6 mm and with a measured composition of 97.10 wt.% Zirconium, 2.12 wt.% Niobium and 0.14 wt.% Tin. Abriata et al. (1986) reported the melting point of pure zirconium to be 1855 °C, while the phase diagrams presented by Lustman and Kerze (1955) indicates that the presence of niobium and tin will reduce this melting point by no more than 20 °C. To prepare the sheets for laser cladding, they were cleaned with ethanol. Then, the side to be cladded was ground using 180 grit silicon carbide (SiC) paper until rolling marks from previous manufacturing process were no longer visible. The sheets were then cleaned with acetone and ethanol.

To prepare the samples for optical microscopy, the clads had to be active polished using a solution of 1 vol.% hydrofluoric acid, 3.5 vol.% nitric acid, 1.5 vol.% hydrogen peroxide and balance water. Extra care was taken to safeguard the researchers and properly dispose of the waste that resulted from sample preparation.