Surface & Coatings Technology 240 (2014) 286–292

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Laser processing of Fe based bulk amorphous alloy coating on zirconium

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ARTICLE INFO

Article history:
Received 27 August 2013
Accepted in revised form 18 December 2013
Available online 29 December 2013

Keywords:
Bulk amorphous alloy
Zr substrate
Laser processing
Coating
Wear

ABSTRACT

Laser Engineered Net Shaping (LENS™), a commercially available laser-based additive manufacturing technique, was employed for the processing of Fe-bulk amorphous alloy (Fe-BAA) coating on zirconium. SEM analysis revealed an amorphous–crystalline mixed phase composite microstructure of the coating. Crystalline phase was dispersed in an amorphous matrix. Moreover, considerable mixing of the Fe BAA in the zirconium substrate was noticed. The Fe BAA coatings showed 800% increase in hardness and decrease in normalized wear volume was ~30 times compared to the control Zr substrate. During the wear testing in 3.5% NaCl solution, Fe BAA coatings showed evidence of fretting corrosion. Overall, our results demonstrate that laser processing can be used to deposit amorphous coatings on Zr substrate.

1. Introduction

Bulk amorphous alloys (BAA) or bulk metallic glasses (BMG) show extraordinary mechanical and electrochemical properties that result from the absence of grain boundaries in the amorphous structures [1–7]. Processing of bulk amorphous alloys is challenging due to the need for a high cooling rates to retain the amorphous phases. Therefore, interest is growing in harnessing properties of this class of materials in the form of coatings on important engineering materials [8–10]. Even then, challenges remain in the areas of retaining the amorphous nature of the coating and creating a strong interfacial bond between the substrate and the coating particularly for high melting point substrates. Apart from those two, some other factors include improving mechanical properties of the coating without compromising the substrate quality, avoiding oxidation of the substrate and BAA during processing, ability to coat large area complex shaped components, and reliability as well as reproducibility of the coatings. The viability of many different processing methods has been investigated in the past. These methods include sputtering [10], copper mold casting [11–14], aluminothermic reaction [15], compression shearing technique [16], rapid quenching from the melt [17,18], plasma processing [19], spraying processing [20,21], spin casting/melt spinning [22–24], microwave processing [25] as well as high power laser processing [26–32]. The simultaneous deposition and melting method can also create amorphous coatings in a single step preventing crystallization of the amorphous precursor powders to the maximum extent with small morphological variations [33]. For coatings of various thicknesses and relatively complex shapes on different metallic substrates, laser-based freeform fabrication technique can be ideally suited for different applications. In the present study, Laser Engineered Net Shaping (LENS™), an example of such a technique, is used for processing of Fe-based bulk amorphous alloy coating on Zr-substrate.

Fe-based bulk amorphous alloys (BAA) have shown excellent mechanical and electrochemical performance [34–44]. In this study, Fe based BAA coatings are deposited on zirconium, a high temperature metal of significant engineering importance due to their different applications in structures demanding corrosion resistance at elevated temperatures. These amorphous coatings on zirconium were processed and characterized for their microstructure, hardness and corrosion behavior under load using a ball-on-disc setup in salt water. Further, a composite zone model is used to show the microstructural evolution of the Fe-BAA coating on Zr during laser processing.

2. Experimental procedure

2.1. Laser processing

Gas atomized NanoSteel NSSHS9172 Super Hard Alloy Steel Powder (from The NanoSteel Company Inc, Idaho Falls, ID) with a nominal composition (atomic %) of $Cr_{<25}$ $Mo_{<6}$ $W_{<15}C_{<4}$ $Mn_{<2}$ $Si_{<2}$ $B_{<5}$ $Nb_{<12}$ $Fe_{balance}$ having particle size distribution between 15 μm and 53 μm was used. The substrate used was a 2 mm thick plate of commercially pure zirconium (Purity level >99%). We have used LENS™ 750 (Optomec Inc., Albuquerque, NM) available in our laboratory to fabricate rectangular shaped samples (6 cm × 1.5 cm) with 0.865 ± 0.080 mm thick BAA coating deposited layer-wise from the feedstock powder. The LENS™ 750 uses a 500 W continuous wave Nd:YAG laser. The Fe BAA powder was fed into the melt zone via delivery nozzles through Argon carrier gas. Laser power for deposition of the Fe BAA powder was set at 450 W, which was needed to melt the Zr-substrate, and the laser scanning speed was 48 in/min (122 cm/min). LENS™ offers consistent and

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0257-8972/$ – see front matter
http://dx.doi.org/10.1016/j.surfcoat.2013.12.043

Table 1
Establishing LENS™ process parameters for processing of Fe BAA coatings on Zr.

Sr no	Processing parameters			Result	Comment
	Laser power	Powder feed rate	Scan speed		
1	375 W	1.2	48 in/min	Insufficient laser power	Unmelted substrate and feedstock powder
2	400 W	1.2	48 in/min	Insufficient laser power	Unmelted feedstock powder
3	400 W	1.2	24 in/min	Increased gas porosity and unmelted particles	Laser power sufficient to just melt the substrate and powder but slower speed gives increased porosity
4	450 W	1.2	48 in/min	Sufficient laser power	Powder melting achieved, good bonding to substrate, top layer slightly unmelted
5	450 W	1.4	48 in/min	Unmelted powder due to higher feed rate, gas porosity	Laser power sufficient but feed rate is too high to give complete powder melting

reliable processing conditions for all samples. The entire assembly is enclosed in a glove box and the oxygen level is constantly monitored by a highly sensitive oxygen sensor, maintaining the environmental O_2 level at than 10 ppm. This limits any possible oxidation and related contamination of the amorphous alloy during the melt cast operation of LENS^{TM} processing.

2.2. Microstructural characterization

The LENS™ processed samples were carefully polished using silicon carbide paper. Final polishing was done using 0.03 μm alumina suspensions on rotating velvet cloth wheel. LENS™ processed Fe BAA coatings on Zr were characterized using optical microscopy and SEM (FEI Quanta 200 and Hitachi S570). XRD analysis was done using a Siemens D 500 Kristalloflex diffractometer with Cu Kα radiation at 20 kV between the 2θ range of 20° and 80° and a Ni filter keeping the step size at 0.1 s.

2.2.1. Samples for corrosion-wear testing

The surface of the coatings was carefully polished to mirror finish prior to wear testing. The BAA coated samples were in 6 cm × 1.5 cm in shape. The coated top surface was wet grinded on 120 grit SiC paper and then subsequently fine grinded till 1200 grit. Final polishing was done using 1, 0.5 and 0.03 micron alumina suspensions in deionized water. Care was taken to keep the coating surface extremely flat throughout the sample preparation process. Before performing wear tests, the samples were ultrasonically cleaned in 50% ethanol solution for 30 min.

2.3. Corrosion-wear and hardness testing

Corrosion-wear testing was done in saline medium (3.5% NaCl) at room temperature on coatings using linear reciprocating wear test with a Nanovea series tribometer. Silicon nitride ball of diameter (Hardness ~1550 $HV_{0.1}$) was used in the tests. Load was kept constant at 7 N, and speed was 1200 mm/min. The amplitude of wear track was 10 mm and tests were performed for a distance of 1 km for all the samples.

Vickers microhardness (Shimadzu, HMV-2 T) tests were performed on the Fe BAA coated Zr and the Zr substrate with a standard diamond Vickers indenter. The load was 100 g (0.98 N) and dwell time was 15 s for all samples. Each reported hardness value is an average of at least 5 similar tests performed on the sample. Variation of hardness with the depth of the coating was recorded by testing the samples in cross section and maintaining equal spacing in between the consecutive tests. General hardness of the Fe BAA coatings was recorded approximately at the center of the cross section.

3. Results

The LENS™ processed Fe BAA coatings on Zr were analyzed using SEM. Wear and wear induced corrosion phenomena were also studied along with hardness measurements.

3.1. Establishing process parameters for processing Fe BAA coatings on Zr

Numerous trials had to be carried out in order to establish the processing parameters of the LENS™ setup for Fe BAA. The most important ones were the laser power; powder feed rate and the scan speed. The various trials are tabulated in Table 1. The effect of every trial was studied using simple optical microscopy and visual examination of the coatings. To adequately melt the Zr substrate and the fine Fe BAA powder, laser power of at least 400 W was necessary. Anything below 400 W could not melt the powder and the substrate; and produced coatings of poor morphology. The powder could not be adequately melted and bonding to the Zr substrate was weak. This caused delaminating or ‘peeling off’ of the BAA coating during grinding and