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Influence of annealing on mechanical and electrochemical properties of cold sprayed niobium coatings

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ABSTRACT

In the present study, thick and dense niobium coatings were obtained using cold spray technique by using air as a process gas. Inter-splat boundaries are completely removed in the coatings heat treated at 1500 °C by the formation of equiaxed grains. Heat treatment reduces the porosity level to ~0.1%. Inter-splat boundary bonding state of the heat treated coatings was investigated using micro-tensile testing, scratch testing and nanoindentation and compared with the bulk niobium. The elastic modulus of the cold spray coatings heat treated at 1500 °C exhibits as high as 103 GPa whereas the same for bulk is 105 GPa. The increase in mechanical strength of inter-splat boundary from *as*-sprayed condition to 1500 °C was estimated to be 750%. Similarly corrosion performance of heat treated coatings was also evaluated in 1 M KOH solution through potentiodynamic polarization as well as impedance spectroscopy studies. The corrosion rate for the coatings heat treated at 1500 °C was estimated to be 0.443 MPY which is comparable for the bulk (0.498 MPY). Coatings annealed at 1250 °C and above, which is very close to the recrystallization temperature of niobium, were found to perform almost as bulk niobium indicating exciting implications for various applications. Assessment of structure–property correlations was done based on the microstructure, porosity and inter-splat bonding state, together with the mechanical and corrosion properties of the heat treated tantalum cold sprayed coatings.

1. Introduction

Cold spraying is a high rate deposition technique to obtain dense metallic coatings with low oxide content, and this low-temperature and high-pressure process has attracted increasing attention over the past two decades [1–5]. Thermo-mechanical characteristics of feedstock and substrate at high strain rates are responsible for bonding and coating build up in this process. Studies have shown that adiabatic heating accompanied by shear instability induces bonding in cold spray deposition [2,3,6]. A variety of materials have been deposited using the cold spray technique for different applications, ranging from hot corrosion [7] to restoration of worn components [8].

The high strength and high melting temperature of body centered cubic (BCC) refractory metals makes them attractive for many applications [9–11]. Among them, niobium possesses a number of interesting properties such as low ductile to brittle transformation temperature, and moderate density. It can be cold worked without intermediate annealing, since the pure niobium is extremely ductile due to its low strain hardening rate [12]. These physical properties play an important role in high strain rate plastic deformation processes such as cold spraying being attractive for niobium processing/deposition. Niobium has an excellent corrosion resistance in all mineral acids except hydrofluoric acid. It is used to fabricate corrosion resistance process equipment including reaction vessels, columns, heat exchangers and thermowells [13]. It is also used in the fabrication of sputtering target in semiconductor industry [12,14]. There is no available scientific data on the deposition characteristics of the niobium using cold spray technique till date.

In cold spraying, coating build up is accompanied by plastic deformation, which is severe at the particle boundaries upon impact. The *as*-sprayed cold spray coatings tend to be brittle due to heavily cold worked splats that form upon impact of feedstock particles and the associated incomplete inter-splat bonding. These imperfections lead to high hardness and low elongation to fracture [15,16] due to the fact that only a fraction of any splat is well bonded with the adjacent splat, leaving the bonding state fragile. A number of defects that are introduced in the coating upon high-velocity impact of feedstock particles due to the anisotropic nature of plastic flow can be healed by a post-coating annealing step. It is widely accepted that heat treatment of cold sprayed coatings enhances the coating properties and performance due to the improved inter-splat bonding that it ensures [17,18]. Many studies have been devoted to ascertain the effect of heat treatment on the functional properties such as corrosion resistance [18,19], electrical conductivity [20] and elastic modulus [21] of cold sprayed coatings. It is now also well proven that the annealing step

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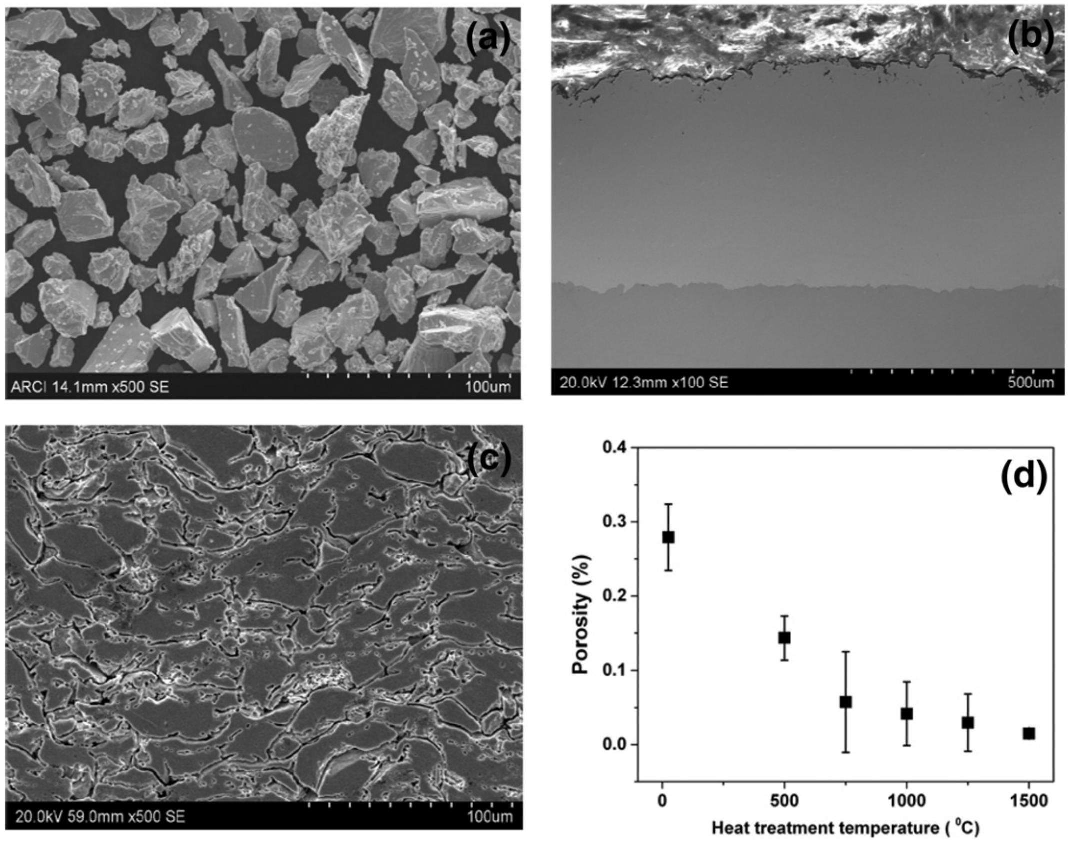

Fig. 1. SEM micrographs of (a) niobium feedstock powder, (b) cross section of the as deposited and (c) etched coating; (d) porosity of the coatings as a function of heat treatment temperature.

induces inter-splat bonding at locations where plastic deformation is severe. Gartner et al [22] and Seo et al [23] have concluded that annealing of cold sprayed coating induces recrystallization, grain growth and diffusion. Seo et al. [23] suggest that annealing of cold sprayed coating at temperatures exceeding the recrystallization temperature leads to grain coarsening which alters the physical and mechanical properties of the coatings. In this study, the influence of annealing temperature on the structure and performance of cold sprayed niobium coating is demonstrated using mechanical and corrosion performance.

2. Experimental details

2.1. Cold spray coatings and heat treatment

A portable cold spray system fabricated in the authors' laboratory was used to deposit coatings using compressed air as both process and powder carrier gas. The high pressure process air was preheated before entering the de Laval nozzle having rectangular exit. The powders were fed into the nozzle through the separate powder feeder line

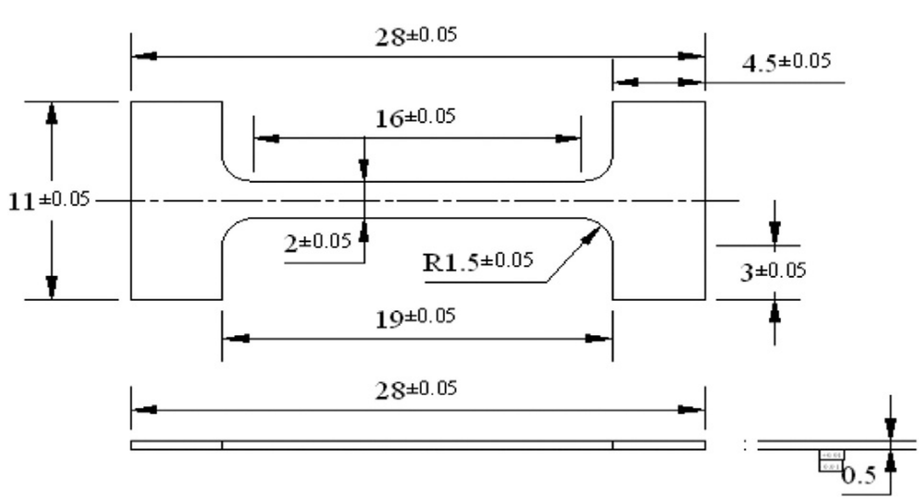

Fig. 2. Dimensions of the microtensile specimen.