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Synthesis and characterization of hard metal coatings by electro-plasma technology

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Abstract

Electro-plasma technology (EPT) is a cathodic atmospheric plasma process which has shown great promise for the deposition of metal coatings exhibiting excellent adhesion to the substrate along with high deposition rates. The present study involves synthesis and characterization of molybdenum coatings using EPT processing. The morphology, composition and structure of Mo-coated surfaces were characterized. The results indicate the successful deposition of molybdenum, with molybdenum alloyed into the surface of both the 4330V steel and Inconel 718 substrates. The surface morphology and roughness of the coated samples reflect unique EPT-induced micro-roughness features. Superposition of the EPT-induced micro-roughness profile on the macro-roughness profile of the substrate surface provides the potential to improve adhesion characteristics of surface. An increase in hardness observed on Mo-coated steel indicates potential to produce hard surfaces by EPT, leading to many advanced applications.

Keywords: Electrolytic plasma; Surface modification; Alloying; Mo coating; XRD

1. Introduction

Electro-plasma technology (EPT) is a hybrid of conventional electrolysis and atmospheric plasma processing [1]. Formation of stable plasma, at atmospheric pressure, on the surface of conductive materials during electrolysis of various aqueous electrolytes gives the capability to conduct various surface treatments including cleaning [2,3], coating [4,5] and surface texturing [6]. The presence of plasma micro-discharges over the work piece (generally the cathode) leads to melting of localized micro-zones on the surface. Subsequent quenching by the liquid electrolyte and mechanical effects produced by imploding plasma bubbles has shown to form a surface with unique characteristics [4–6]. Furthermore, surface interactions during the non-equilibrium EPT processing provide a great potential to alloy various elements into metal surfaces.

Mo coatings have gained attention due to their application in various industries such as aerospace, chemical, pulp-and-paper, etc. Mo coatings are also potential candidates for replacement of Cr as a protective coating. The most common deposition method for Mo coatings is thermal plasma spray [7–9]. Thermally sprayed coatings are usually inhomogeneous and discontinuous, characterized by pores, oxide lamellas or partly molten spray particles [8]. Mo coatings generally exhibit a layered structure [7,8]. It has been reported that the oxide phase MoO_2 is present between the layers, which may reduce the interlameller strength [7]. There is a need for new technologies for the deposition of Mo, and EPT processing may provide such capability. It is interesting to note that the thermal spray process, prior to coating, requires grit blasting in order to clean and obtain necessary surface roughness. EPT has shown to clean metal surfaces with the creation of uniform micro-roughness and morphology that provides better coating adhesion as compared to grit blasting [2,10].

Electrodeposition of Mo coatings from aqueous electrolytes has not been very successful, but its co-deposition

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has been investigated in the past [11,12]. To the best knowledge of the authors, there is no literature on the study of deposition of Mo coatings by electro-plasma processes. In the present study, Mo coatings were deposited on 4330V steel and Inconel 718, using EPT. Surface characterization and structure analysis of the Mo coatings are presented.

2. Experimental details

The present work involved deposition of Mo coatings by EPT on 4330V and Inconel 718 substrate materials. The same coating procedure was used for all samples. Experimental details associated with the EPT process have been described in detail elsewhere [4,6]. Mo coatings were deposited using a solution of sodium molybdate (Na_2MoO_4), with additional Mo supplied to the solution in the form of Mo powder. The processing conditions were set to achieve a power density of 139.5 W/cm^2 and a processing time of 5 min was used for all specimens. The differences among the samples lay in the substrate material and the surface preparation prior to coating. The following substrates were used—4330V steel (samples Mo-S1 and Mo-S2) and Inconel 718 (samples Mo-I1 and Mo-I2). Test coupons were cut in rectangular pieces with dimensions of 10 cm × 2.5 cm × 2.5 cm. Surface preparation options included polishing by metallography and cleaning by EPT processing. Samples Mo-S1, Mo-S2 and Mo-I1 were polished to a mirror finish (to 0.05 μm), prior to coating. Samples Mo-S1, Mo-I1, and Mo-I2 were cleaned by EPT processing for ~20 sec using sodium bicarbonate based electrolyte prior to coating. One sample of In718 (Mo-I2) was coated with Mo in as-received state (without polishing) after EPT cleaning. The sample preparations are summarized in Table 1.

The surface morphology and composition were analyzed using a Hitachi S-4500II field emission scanning electron microscope (SEM) with energy-dispersive X-ray spectroscopy (EDX). Surface roughness and profile of the samples was measured using an optical profilometer (Wyko NT-3300). Average roughness (R_a), rms value of roughness (R_q) and total roughness (R_t, i.e. the sum of vertical distances from the deepest valley and to the highest peak) values are reported. Roughness parameters reported represent the average of three scans taken at different places on the processed sample surfaces.

Table 1
Surface preparation steps prior to Mo coatings

Sample	Substrate	Polished	EPT-cleaned
Mo-S1	4330V	Yes	Yes
Mo-S2	4330V	Yes	–
Mo-I1	In718	Yes	Yes
Mo-I2	In718	–	Yes

X-ray diffraction measurements were performed using a Panalytical X'Pert Pro system using Cu K_α radiation with an operating voltage of 45 kV and current of 40 mA. Continuous scans were performed from 30° to 90° 2θ with a 0.03° step size and a counting time of 4 sec/step. Scans were made in a standard Bragg–Brentano (θ–2θ) mode, as well as in grazing incidence mode with a fixed incidence angle of 2° for enhanced surface sensitivity. This results in a sub-micron X-ray attenuation length for 8.04 keV (Cu K_α) radiation (~0.14 μm for pure Fe; 0.22 μm for pure Mo), where attenuation length is defined as the depth into the material measured along the surface normal where the intensity of X-rays falls to 1/e of its value at the surface [13].

The hardness of Mo-coated surface was determined with a Zeiss microhardness tester. Multiple indentations were carried out at a load of 50 g using a Knoop indenter. The microhardness reported is the average of at least five indentations.

3. Results

3.1. Surface morphology

Fig. 1 presents micrographs obtained from the two coatings deposited on 4330V steel substrates—Mo-S1 and Mo-S2. These images reveal surfaces that are relatively flat, reflecting the use of a polished substrate, but exhibit a morphology characteristic of electro-plasma processing [2–6]. This morphology is distinguished by the formation of micro-craters and spheroid-shaped elevations, which results in creation of the micro-roughness. This morphology is uniformly distributed across the surface of sample Mo-S1, as shown in Fig. 1(a) and (b). In specimen Mo-S2, this EPT morphology is interspersed with flat featureless regions, as shown Fig. 1(c) and (d). The difference between these two specimens is that the specimen Mo-S1 was cleaned using the EPT process prior to the EPT coating step, while the specimen Mo-S2 was directly subjected to the coating step. This indicates that the inclusion of the cleaning step (or increased total EPT processing time) provided a more homogeneous morphology of the EPT-modified surface. The scale of the EPT-produced morphological features can be estimated from these images. In both the Mo-S1 and Mo-S2 surfaces, the typical diameter of the micro-craters is estimated to be of the order of 3–5 μm.

Typical SEM micrographs obtained from the two coatings deposited on Inconel 718 substrates – Mo-I1 and Mo-I2 – are presented in Fig. 2, along with the images obtained from the as-received substrate material. The SEM images of Mo-I1 (polished and EPT-cleaned prior to coating) shown in Fig. 2(a) and (b) reveal the characteristic EPT induced morphology evenly distributed over an otherwise flat surface. The uniform distribution of the surface morphology is similar to that observed for sample Mo-S1, which was