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Full Length Article

The microstructure and properties of tungsten alloying layer on copper by high-current pulse electron beam

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ABSTRACT

In this paper, tungsten (W) was chosen to be an alloying element into the surface layer of commercial pure (cp) copper (Cu) to enhance the surface properties after surface alloying by high current pulsed electron beam (HCPEB). After HCPEB irradiation, part of the blended powder was dissolved into the substrate surface to form an alloyed layer, which has defect structures consisting of dislocation cells/walls. The presence of defect structures induced by HCPEB irradiation provides a large amount of paths for W atoms to form solid solution and ultrafine W particles. The surface hardening is ascribed to intense plastic deformation *via* stress, sub-grain strengthening, along with the alloying elements via precipitation hardening and/or the solid solution strengthening. The corrosion performance, tested in 3.5% NaCl solution, was significantly enhanced after surface alloying. The improvement in corrosion resistance is essentially attributed to the combination of the structure defects and the addition of alloying elements to form more stable passive film.

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1. Introduction

Because of its highly electrical and thermal conductive, malleable and ductile, easy to manufacture and relative cheap, copper (Cu) and its alloys have been extensively used as the electric contact material [1–3]. However, those shortcomings, which are soft, susceptible to wear, and prone to corrosion in chloride-containing environment, impede the further widespread use. An ideal coating deposited by sputter/vapor on the substrate was introduced, which includes such properties: good physical and chemical compatibility with the substrate, resistance to an aggressive environment in the presence of Cl^- ions solution. However, the coating suffers from low binding between the coating and substrate. Therefore, the enhanced alloying layer can be fabricated by a surface alloying tool capable of processing a coating/substrate system (film deposited on a substrate) [4–7]. Tungsten (W) is an immiscible element employed in Cu composite material for its high-temperature hardness, strength and high arc erosion resistance. Previous studies have shown that the addition of W into the alloys can efficiently enhance corrosion resistance by the formation of the stable passivity [5,8]. Considering the above factors, the surface alloying with W element should be an efficient method to improve the mechanical properties of the matrix material without changing the substrate properties.

As surface modifying techniques, featuring with the microstructure optimization, ion, laser and electron beam have received an increasing attention. However, among various surface treated techniques, high-current pulsed electron beam (HCPEB) has drawn increasing interests in recent years due to its capability, which not only just overcomes the problem, impact of ionic impurities caused by ion beam irradiation, but its energy efficiency is much higher than that of the laser beam. The pulsed electron irradiation produces rapid heating, melting and rapid solidification of the material surface, which makes it a good choice for modifying and alloying of material surfaces [9–11]. In this process, an alloyed layer of the substrate (∼several micrometers) by components of the coating can be achieved, with solid solution and chemical compounds being formed between elements of the substrate and the coating [12–14]. In addition, the non-equilibrium temperature field coupled together with dynamic stress field were induced by the extremely rapid thermo cycles of HCPEB irradiation, resulting in the formation of non-equilibrium microstructures, such as vacancies, dislocations, and stacking faults [15,16]. Abundant defect structures provide a great amount of diffusion paths for immiscible atoms due to the low activation energy demanded for diffusion, which results in that the immiscible atoms tend to strongly segre-

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gate to defect structures/grain boundaries (GBs). These effects can be especially important for grain stability due to the pinning effect to lead to nano-crystalline materials (resistance to grain deformation evenly at elevated temperatures) [17], which demonstrates a drastically improved mechanical properties. Pogrebnjak et al. found that the surface hardness and corrosion resistance of a mixed layer of Ta/Fe system after irradiation were enhanced compared with the initial properties of a-Fe [18]. It was also observed that the corrosion resistance could be improved in proper parameters for Ti surface alloying of an AISI 316L stainless steel due to the homogeneity of Ti distribution [9]. Hence, previous researches indicated that the process of surface alloying induced by HCPEB irradiation was a possible way to enhance surface properties, which unable to implement by conventional surface treatment techniques.

In this work, in order to enhance the surface properties in the extreme condition, W was added into the surface layer of Cu by using a duplex processing, coating deposition followed by HCPEB irradiation. The phase composition, surface morphology, microstructure and properties of the surface alloying layer were characterized for investigating the mechanism behind it.

2. Experimental procedures

The as-received commercial pure (cp) Cu was machined into square plates with dimensions of 10 × 10 × 5 mm. All the samples were grounded using SiC abrasive papers, polished with diamond paste (1 μ m) and cleaned ultrasonically in acetone. The mixture of 85 wt% of Cu powder (99.95% in purity with 45 μ m particle size) and 15 wt% of W powder (99.99% in purity with 5–10 μ m particle size) was blended for 5 h in ball mill, which exhibited a spherical particles with about 10–50 nm in size, as shown in Fig. 1(a), and the corresponding Selected Area Electron Diffraction (SAED) was shown in Fig. 1(b). A slurry was prepared by mixing 3 g of the blended mixture and organic binder (100 mL) (Nitrocellulose lacquer:diluent is 1:2), which was sprayed onto Cu samples using an air-pressurized spraying gun. Prior to the spraying, the polished surfaces of samples were irradiated at room temperature with 10 pulses using HOPE-I type HCPEB source, which is a modification of Nadezhda-II E-beam machine [19] (developed at the Institute of High Current Electronics, Russia), under the following parameters: the electron energy 27 keV, the current pulse duration 1.5 μs, the energy density 4 J/cm^2, the beam diameter 60 mm, and the vacuum 5.0×10^{-3} Pa. Subsequently, the surface alloying process was carried out with 20 and 30 pulses under above mentioned parameters.

A X-ray diffraction (XRD) with CuKα radiation in a Rigaku D/max-2500/pc X-ray diffractometer was adopted for phase identification. The microstructure evolution of the overlay was comprehensively studied by using a JEOL JSM-7100F scanning electron microscope (SEM) with an Inca energy 350 energy dispersive spectrometer (EDS) at 15 kV accelerating voltage. In order to clarify the resultant phase and the induced microstructure modifications within the alloying layer, the thin foils samples were prepared by mechanical pre-thinning, dimpling, and jet electrolytical thinning from the substrate side for transmission electron microscope (TEM) observation, and the TEM observation was conducted with a JEM-2100 high resolution microscope.

Microhardness was measured by a HVS-1000 device with a load of 0.245 N (25 g) applied for 15 s. Five of seven reading were averaged to reduce random error, (the maximum and minimum were omitted). A CHI660C electrochemical workstation was implemented. The whole set-up was connected to a conventional three-electrode cell, including a work electrode, a saturated calomel electrode (SCE) as the reference electrode and a platinum sheet as the counter electrode and the electrolyte solution was 3.5 wt% NaCl (0.6 M). After samples exposed to electrolyte solution under open circuit potential at room temperature for 30 min, standard potentiodynamic polarization and electrochemical impedance spectroscopy measurements were performed. The cyclic polarization (CP) was done on at a sweep rate of 0.333 mV/s, and the electrochemical impedance spectra (EIS) was obtained in the frequency between 10^{-2} and 10^5 Hz at the open circuit potential (OCP) with the excitation signal of 10 mV in amplitude. The test specimen is used as a working electrode, and was exposed to the solution to an area of 1 cm^2. At least three readings were obtained to ensure the consistency of CP results.

3. Results

3.1. Surface microstructures

Fig. 2(a) shows the XRD patterns of samples with different pulses number. For the sample with W coating, the peaks of Cu and W phase were present. After surface alloying, a little shift of Cu diffraction peaks to low angle was present, which is sensitive to a given change in interplanar spacing (d-spacing). The relationship between the Cu lattice parameter (d-spacing parameter) and the HCPEB pulses is shown in Fig. 2(b). The values of Cu lattice parameter (a_{Cu}) increase as the increase of pulse number. Miedema reported that the chemical mixed enthalpy (ΔH_{mix} (Cu → W)) is 86 kJ/mol as Cu dissolves into the W lattice; on the contrary, ΔH_{mix} (W → Cu) is 107 kJ/mol [20]. It is indicated that Cu atoms preferentially dissolve into the W lattice, while W in the Cu lattice is impossible. However, many researchers reported that Cu (W) solid solution was prepared by non-equilibrium method in Cu-rich zone [21–23]. Therefore, the increment in a_{Cu} is attributed to that some Cu atoms were substituted by W atoms to form substi-

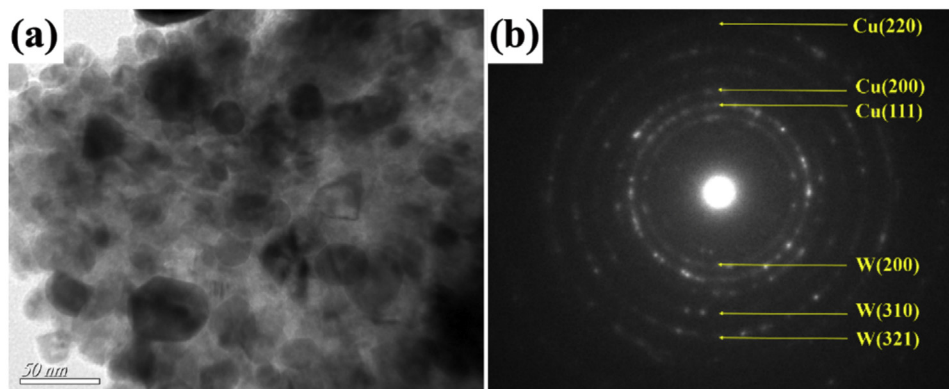

Fig. 1. The TEM image of powder milled for 5 h. (a) The mixture of Cu and W, (b) The SAED pattern

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