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Thermal stability of electrodeposited nanocrystalline nickel assisted by flexible friction

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Abstract: Nanocrystalline nickel coating was prepared by flexible friction assisted electrodeposition technology in an additive-free Watts bath. The coating consists of massive equiaxial crystals with an average grain size of about 24 nm and exhibits a (111) preferred orientation. The differential scanning calorimetry (DSC) analysis of nanocrystalline nickel demonstrates that the peak temperature of rapid grain growth is about 285.4 °C, and the peak temperature of grain growth towards equilibrium is around 431.5 °C. The isochronous annealing results reveal that abnormal grain growth behavior is not observed in nanocrystalline nickel without sulfur-containing. The thermal stability of the deposition was improved due to its initial microstructure of the as-deposited nickel and a certain amount of annealing nano-twins with low-energy, which reduces the driving force for grain growth. Consequently, the coating shows a low residual tensile stress of about 50 MPa and a high microhardness of HV 400 at the annealing temperature of 450 °C.

Key words: electrodeposition; nanocrystalline nickel; flexible friction; thermal stability; grain growth

1 Introduction

Nanocrystalline materials, e.g., nanocrystalline nickel, have been conducted extensive researches and received much attentions because of their excellent physical, chemical and mechanical properties, and some of them have been applied to the engineering [1−4]. However, like other nanocrystalline materials, nanocrystalline nickel is in a thermodynamic non-equilibrium due to its large excess free energy associated with the high volume fraction of grain boundaries. The large excess free energy provides a main driving for thermal [5−7] or stress induced [8,9] grain growth, which significantly reduces the size-dependent properties of nanocrystalline nickel, such as its high hardness [10,11], tensile strength [12], anti-fatigue [13], wear resistance [14]. Therefore, the thermal stability of nanocrystalline nickel has become an important issue in the theoretical and practical application fields.

The thermal stability of nanocrystalline nickel is affected by many factors. These factors include the initial grain size [15], grain shape and its distribution [16], crystalline texture [17], type and concentration of impurities [18], grain boundary structure (smooth or roughness) [19], structural defects (dislocations, twins, etc) [20] and preparation method [16,20]. Although a lot of researches have been done, many aspects are not yet fully understood, e.g., abnormal grain growth behavior. The electrodeposited nanocrystalline nickel coating has a dense and relatively simple structure. Thus, it provides a great convenience for thermal stability study. However, the previous nanocrystalline nickel coating for thermal stability study contains sulfur and carbon impurities owing to the use of additives, such as saccharin [18,21]. This leads to a continuous segregation of solid sulfur along the grain boundary during the thermal annealing process, and promotes the abnormal grain growth and/or accelerates the grain growth after segregation [21−24]. In addition, these inclusions may increase the brittleness, and decrease the corrosion resistance of deposit [25,26].

To inhibit grain growth, a physical friction way with

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flexible media instead of chemical additives was used in this paper. The nanocrystalline nickel scarcely sulfur-containing is prepared by the flexible friction assisted electrodeposition method, and it exhibits massive equiaxial crystals and a (111) preferred orientation. The main purpose of this study includes two aspects: on one hand, it enriches the existing thermal stability knowledge of electrodeposited nanocrystalline nickel as compared with the previous nanocrystalline nickel sulfur-containing, and reveals the role of sulfur impurity; on the other hand, the thermal stability of the deposit is improved by designing the starting nanocrystalline microstructure.

2 Experimental

Nanocrystalline nickel coating was prepared by flexible friction assisted electrodeposition technology in a Watts bath without any additives. The experimental apparatus, matrix material, process, bath composition, etc, were described in Ref. [27]. However, bristles were used as the flexible friction media. The relative moving velocity was 12 m/min.

The differential scanning calorimetry (DSC) curve of nanocrystalline nickel assisted by flexible friction was obtained using an SDT−Q600 simultaneous thermal analyzer. The as-deposited sample was heated at a heating rate of 10 °C/min from the room temperature to 500 °C under the protective atmosphere of argon gas. According to the preliminary results of the DSC analysis, the annealing treatments of the nanocrystalline nickel coatings were conducted at 150, 300, 450 °C for 1 h in a box-type resistance furnace, respectively, and the samples were subsequently cooled in air. In order to analyze the preliminary grain growth, the surface morphology of the coating was observed by a Philips Quanta 200 scanning electron microscope (SEM) at different annealing temperatures. The initial as-deposited structure and the annealing texture evolution of the coating was analysed by a D8 advance multi-crystal X-ray diffractometer (XRD). Furthermore, according to the Scherrer equation and the full width at half maximum (FWHM), the variation of grain size was qualitatively assessed. The grain sizes, grain shapes and their distributions after the heating were observed using a Tecnai G^2 F30 transmission electron microscope (TEM) in order to further confirm the grain growth. The residual stress and microhardness of nickel coating was measured by an X−350A X-ray stress diffractometer and HVS−1000 digital microhardness tester, respectively. The measurement method and specific operation parameters were given in Ref. [27].

3 Results and discussion

3.1 Initial as-deposited microstructure

Figure 1 shows the XRD pattern of the as-deposited nickel coating assisted by flexible friction. It can be clearly seen from Fig. 1 that, the coating shows the strongest diffraction intensity in the (111) plane. Meanwhile, compared with the standard diffraction intensity of nickel powder, it is not difficult to determine a (111) preferred orientation and a face-centered cubic structure in the as-deposited nickel coating assisted by flexible friction.

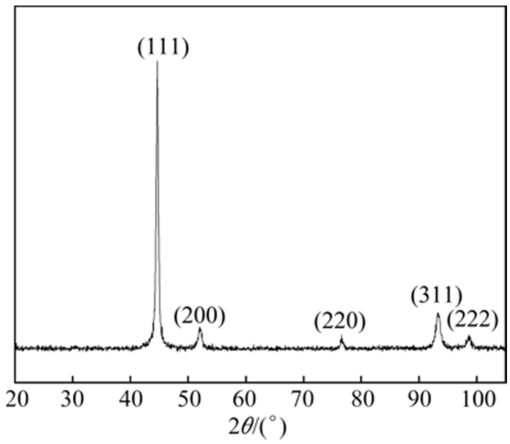

Fig. 1 XRD pattern of as-deposited nickel assisted by flexible friction

Figure 2 exhibits the bright-field TEM image and corresponding selected area electron diffraction pattern, the dark-field image and the grain size distributions of the as-deposited nickel assisted by flexible friction. As shown in Fig. 2(a), the as-deposited nickel coating has a typical polycrystalline structure. This structure is composed of many fine equiaxed crystals, and the grain size distributions range from 10 to 70 nm, and the average grain size is of about 24 nm (Fig. 2(d)). Moreover, it can be observed from Fig. 2(c) that, the coating displays a certain number of nano-twins with different lamellar thickness, and the Moiré fringing phenomenon indicates that a high residual stress exists in the electrodeposited nanocrystalline nickel assisted by flexible friction. Furthermore, the nickel coating has a layered structure. The reason lies in the fact that the periodic flexible friction at the surface of cathode can inhibit the growth of columnar crystals in the direction perpendicular to the substrate and promote the lateral discharging growth of metal nickel ions.

3.2 Differential scanning calorimetry (DSC) analysis

Figure 3 shows the DSC curve of the electrodeposited nanocrystalline nickel assisted by flexible friction. As seen from Fig. 3, one small and one