



## 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

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<sup>2</sup> 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.

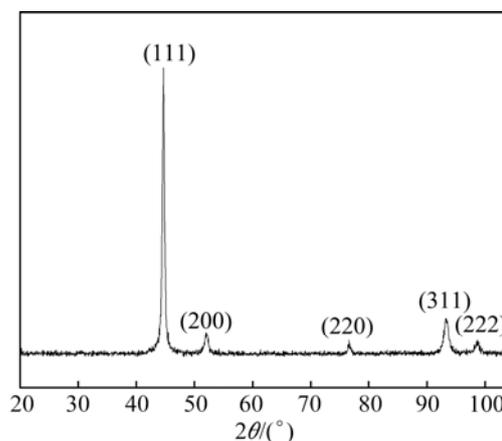
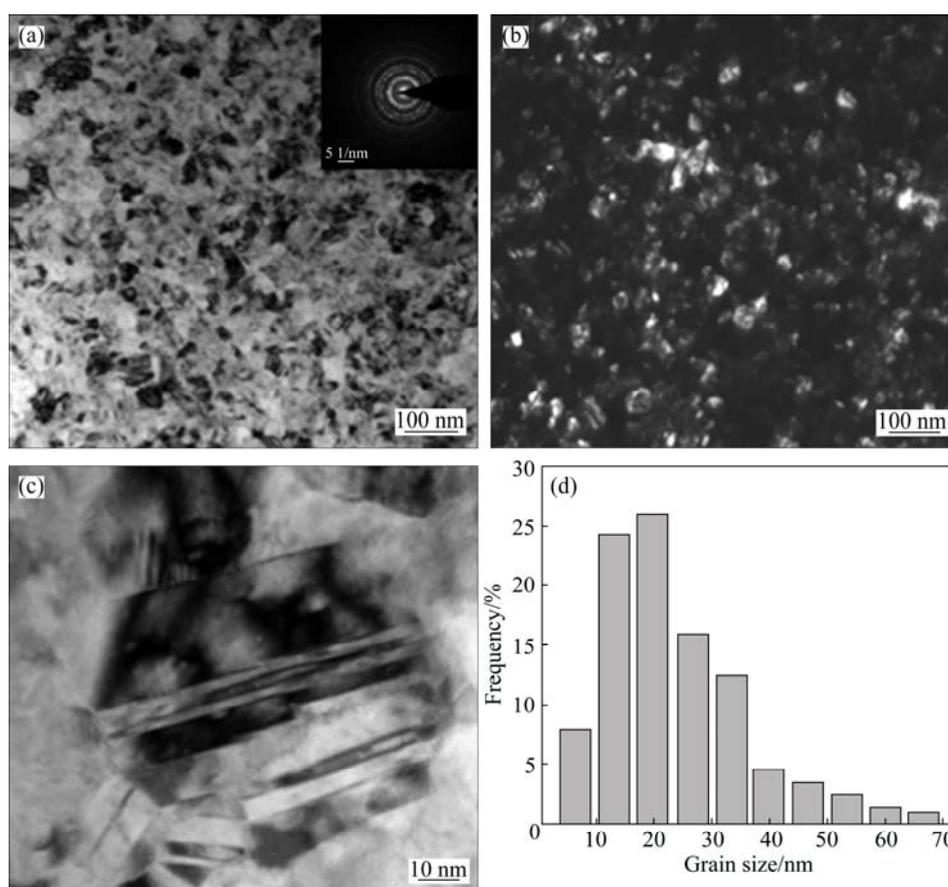


Fig. 1 XRD pattern of as-deposited nickel assisted by flexible friction

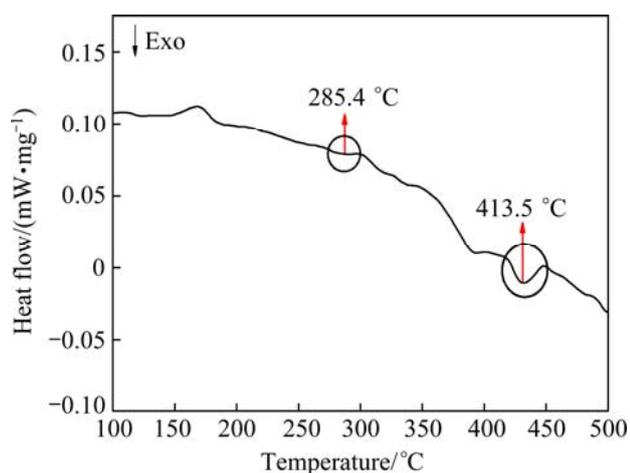
Figure 2 exhibits the bright-field TEM image and corresponding selected area electron diffraction pattern, the dark-field image and the grain size distributions of the as-deposited nickel assisted by flexible friction. As shown in Fig. 2(a), the as-deposited nickel coating has a typical polycrystalline 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



**Fig. 2** Bright-field image and corresponding selected area electron diffraction pattern (a,c), dark-field image (b), and grain size distribution (d) of as-deposited nickel assisted by flexible friction



**Fig. 3** DSC curve of electrodeposited nanocrystalline nickel assisted by flexible friction

big exothermic peak temperatures are at about 285.4 and 431.5 °C, respectively. The former exothermic peak is not obvious, and the peak temperature is between 260–300 °C [5,18,28]. The corresponding exothermic peak indicates the normal or rapid grain growth of nanocrystalline nickel. The electrodeposited nanocrystalline nickel assisted by flexible friction

exhibits almost the same exothermic peak, but much smaller exothermic enthalpy, as compared with the electrodeposited nanocrystalline nickel sulfur-containing with the average grain size of 15–30 nm. The discrepancy stems from the formation of  $\text{Ni}_3\text{S}_2$  compounds in the grain boundary due to the segregation of solid solution, and the released energy of the formation of this compound is much higher than that of the rapid grain growth [23], which may explain the small and unremarkable peak area for rapid grain growth in this study. The latter exothermic peak clearly shows that the peak temperature is between 370–500 °C, which corresponds to faster grain growth and grain growth towards equilibrium [5]. This behavior seems not to show much difference in contrast to nanocrystalline nickel sulfur-containing. However, the exothermic peak of a low temperature nucleation or abnormal grain growth is not observed due to the lack of sulfur-containing additives used for nanocrystalline nickel. Therefore, it can be concluded that the presence of sulfur does affect the grain growth behavior of nanocrystalline nickel, but the thermal stability of the electrodeposited nanocrystalline assisted by flexible friction is not significantly reduced.

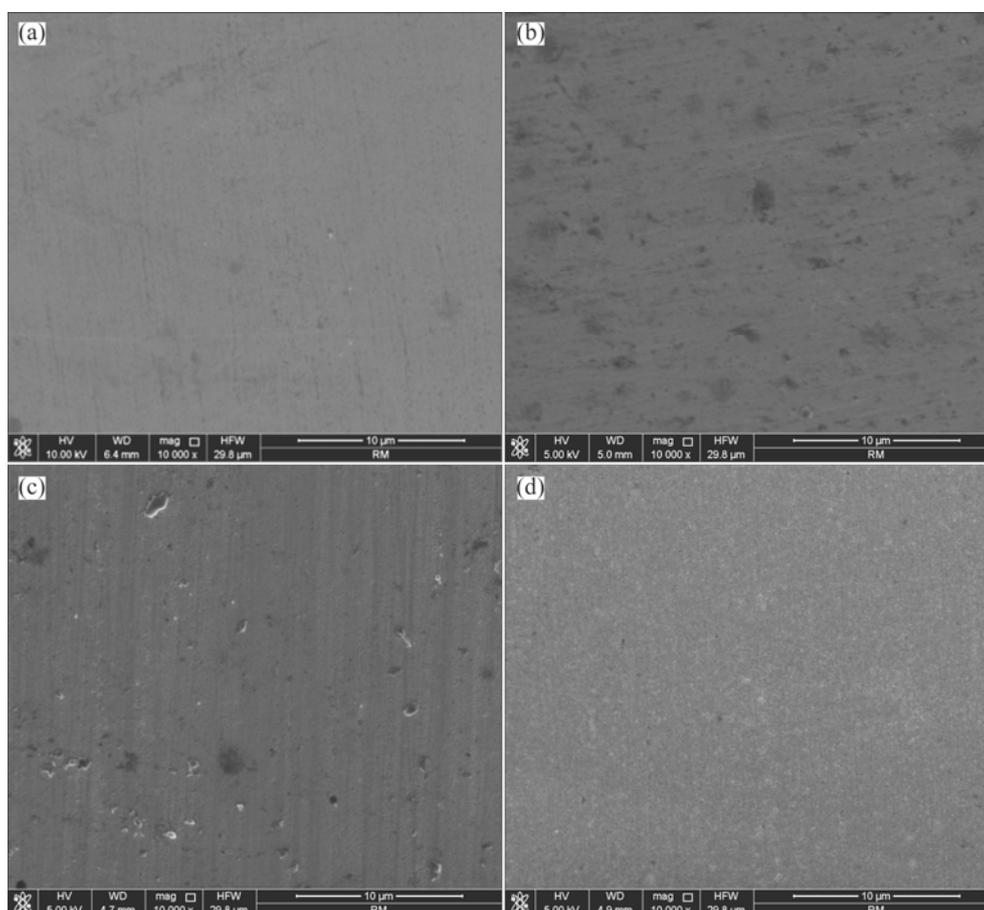
### 3.3 Preliminary grain growth analysis

Figure 4 shows the surface morphology of the electrodeposited nanocrystalline nickel assisted by flexible friction at different annealing temperatures. As can be seen from Fig. 4, the surface morphologies of the annealed nickel coating at temperature of 150 °C and the as-deposited nickel coating have no significant differences, and the two surfaces are of the “brush marks” leaved by flexible friction. The grain size of the both coatings is so small that it is difficult to distinguish through the surface morphology. At the annealing temperature of 300 °C, a large number of small grains grow in the grooves of “brush marks”. These positions belong to a lower energy system, which is helpful for the recrystallization nucleation and fast grain growth of nanocrystalline nickel, but the grain is not completely grown. At the annealing temperature of 450 °C, the surface of the coating is uniformly covered with the spherical particles, and the particle size remains quite small. This indicates that the thermal stability of nanocrystalline nickel is not very low. The reason is that the electrodeposited nanocrystalline nickel assisted by flexible friction has a (111) preferred orientation, and contains a large number of equiaxed crystals and a number of nano-twinning texture. These structural

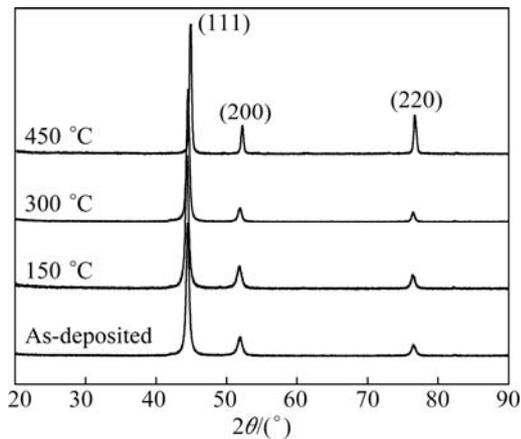
features improve its thermal stability due to the low-energy thermodynamic state and a small driving force for grain growth.

### 3.4 In-depth microstructure evolution analysis

Figure 5 shows the XRD patterns of the electrodeposited nickel assisted by flexible friction at different annealing temperatures. As can be seen from Fig. 5, when the annealing temperature does not exceed 150 °C, the diffraction intensity and FWHM of each crystal face are the same in the annealed and as-deposited nickel coatings. This indicates that grain growth does not occur, and the (111) preferred orientation has no variation. When the annealing temperature reaches 300 °C, the diffraction peak intensity and FWHM of (200) and (220) decrease, which indicates that the grain size of nanocrystalline nickel grows, but the preferred orientation is still (111). When the annealing temperature increases to 450 °C, the diffraction peak intensity, and FWHM of (111) reduce at the same time, and the grain size continues to grow. The diffraction peak intensity of (200) and (220) is relatively enhanced. It indicates that the preferred orientation degree in corresponding crystal plane increases. Finally, the preferred orientation of the annealed coating is (220).



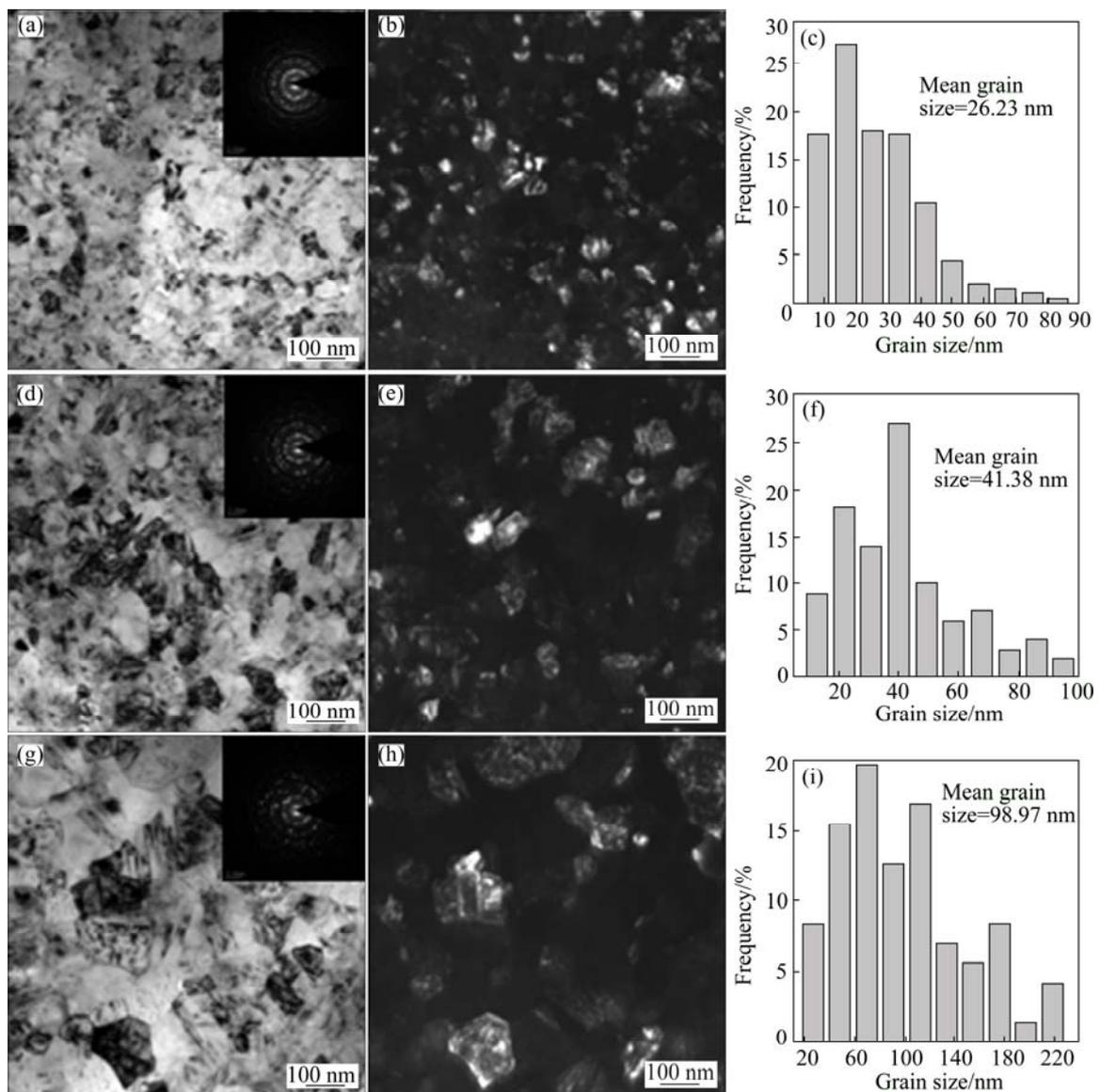
**Fig. 4** Surface morphologies of electrodeposited nanocrystalline nickel assisted by flexible friction at different annealing temperatures: (a) As-deposited; (b) 150 °C; (c) 300 °C; (d) 450 °C



**Fig. 5** XRD patterns of electrodeposited nickel assisted by flexible friction at different annealing temperatures

For a face-centered cubic metal nickel, the surface energy orders of each plane from high to low are (220) > (200) > (111). With increasing the annealing temperature, the energy barrier is continually overcome, and the grain growth towards the direction with a stable structure is at a fast-growing rate. The grain growth rate in high-energy crystal face is accelerated, thus the (220) preferred orientation appears at high-temperature annealing.

Figure 6 shows the TEM images and grain size distributions of the electrodeposited nickel assisted by flexible friction at different annealing temperatures. With increasing the annealing temperature, the abnormal grain growth is not observed in the nanocrystalline nickel, but the grain size gradually increases, which is confirmed by the variational selected area electron diffraction patterns and TEM images. Moreover, the grain size distributions



**Fig. 6** TEM images and grain size distributions of electrodeposited nickel assisted by flexible friction at 150 °C (a–c), 300 °C (d–f) and 450 °C (g–i): Bright-field image and corresponding selected area electron diffraction pattern (a, d, g); Dark-field image (b, e, h); Grain size distributions (c, f, i)

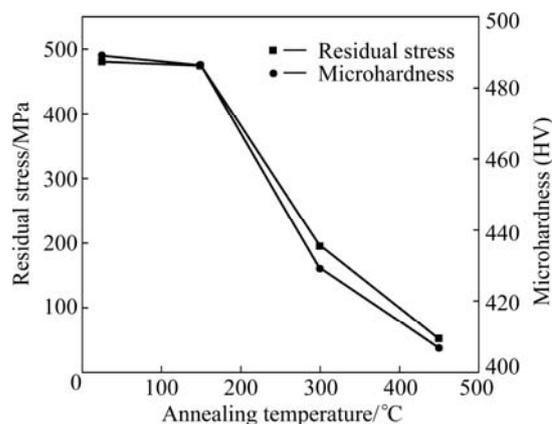
gradually become wider, and the grain shapes also take place evolution. When the annealing temperature does not exceed 150 °C, the average grain size of the nanocrystalline nickel maintains at 23.5–26.2 nm, and the grain growth scarcely occurs. When the annealing temperature reaches 300 °C, the starting grains rapidly grow to the average grain size of 41.3 nm, and the maximum value is close to 99 nm. The crystal shape changes from the equiaxed to the hexagonal lump. When the annealing temperature reaches 450 °C, a large number of annealing twins form, and the grain shape evolves into a six-sided block. The volume of grain boundary and the excess free energy stored in the grain boundaries rapidly decrease. The grain grows towards equilibrium, and the average and the maximum grain size is about 100 and 220 nm, respectively. The TEM analysis for grain growth of nanocrystalline nickel is consistent with the results of DSC, SEM and XRD at different annealing temperatures. Compared with the reported result in Ref. [22], the electrodeposited nanocrystalline nickel with an initial grain size of about 20 nm was annealed at 300 °C, and the average grain size was beyond 100 nm. However, the average grain size of the electrodeposited nanocrystalline nickel assisted by flexible friction is still in the nano-scale at the annealing temperature of 450 °C. This indicates that the thermal stability of the electrodeposited nanocrystalline nickel assisted by flexible friction is improved.

The elevated thermal stability of the nanocrystalline nickel coating lies in the existence of the equiaxed grains, initial (111) preferred orientation, nano-twinning and lamellar structure. The equiaxed grains with high angle boundaries are highly stable and resistant to grain coarsening at high temperatures. The existence of equiaxed structure leads to a decrease of spread of local curvatures of the grain boundary with low energy. Therefore, it is possible to maintain a slow growth under experiencing the grain instability, and the structure is stabilized by a spatially homogeneous distribution of the boundary curvature and energy [16]. A staking structure of nanocrystalline nickel with a (111) preferred orientation and nano-twin defects is in a thermodynamic low energy state, and the driving force for grain growth is small. Moreover, the orientation pinning due to the strong textured lamellae, as well as the suppressed recrystallization nucleation kinetics by the laminated structure in extremely small dimensions, may also contribute to the elevated stability [17]. These analyses are confirmed by the results of DSC and TEM.

### 3.5 Residual stress and microhardness variation

Figure 7 shows the residual stress and microhardness of electrodeposited nickel assisted by flexible friction at different annealing temperatures. It can be

seen from Fig. 7 that, when the annealing temperature does not exceed 150 °C, the residual tensile stress and microhardness of the nanocrystalline nickel coating have little variation. This suggests that the grains do not grow, and the atoms of grain boundaries do not rearrange or relax. When the annealing temperature is higher than 150 °C, with increasing the annealing temperature, the atoms in the grain boundaries of nanocrystalline nickel rearrange orderly, and the grains grow rapidly. The relaxation process of grain boundary in nanocrystalline nickel always occurs prior to or simultaneously with the grain growth process, which leads to the decrease of the residual tensile stress and microhardness of the coating. When the annealing temperature reaches 450 °C, the grains grow more quickly, and the residual tensile stress reduces from 481 MPa for the as-deposited nickel to about 50 MPa at the annealing state which is close to the stress equilibrium of the coating. The microhardness reduces from about HV 500 for the as-deposited nickel to about HV 400 at the annealing state, but the hardness remains high.



**Fig. 7** Residual stress and microhardness of electrodeposited nickel assisted by flexible friction at different annealing temperatures

In combination with the TEM analysis of electrodeposited nanocrystalline nickel assisted by flexible friction annealed at 300–450 °C, the crystal grains grow rapidly and the residual tensile stress of the coating releases quickly. A certain amount of annealing nano-twins form (Figs. 6(d) and (g)), and these annealing nano-twins reduce the grain boundary energy and the driving force for grain growth. Furthermore, these nano-twins interact with dislocations. Thus, the thermal stability and microhardness of the nickel coating is still high.

## 4 Conclusions

1) Nanocrystalline nickel coating without sulfur-containing was prepared by the flexible friction assisted

electrodeposition technology in the Watts bath without any additives, and the coating exhibits a (111) preferred orientation and the equiaxial grains with an average grain size of about 24 nm.

2) At different annealing temperatures, abnormal grain growth behavior for the nanocrystalline nickel coating is not observed. The thermal stability of the coating is improved due to the initial as-deposited structure and a certain amount of annealing nano-twins, which reduces the driving force for grain growth.

3) The annealing temperature has an effect on the structure, residual tensile stress and microhardness of the electrodeposited nanocrystalline nickel assisted by flexible friction. When the annealing temperature does not exceed 150 °C, the structure, residual tensile stress and microhardness of the annealed nickel are the same as those of the as-deposited nickel. At the annealing temperature of 300 °C, the grain size grows rapidly, the residue tensile stress and microhardness decline, but the coating is still a (111) preferred orientation. When the annealing temperature reaches 450 °C, the preferred orientation of the coating transforms into (220), the grain size further increases towards equilibrium, and the tensile residual stress and the microhardness further reduce to approximately 50 MPa and HV 400, respectively.

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## 柔性摩擦辅助电沉积纳米晶镍的热稳定性

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**摘 要:** 采用柔性摩擦辅助电沉积技术在无添加剂的 Watts 液中制备(111)择优取向且平均晶粒尺寸约为 24 nm 的等轴纳米晶镍镀层。差示扫描热分析结果表明: 该纳米晶镍镀层的快速晶粒生长峰值温度约为 285.4 °C, 晶粒生长趋于平衡峰值温度约为 431.5 °C。等时退火结果表明: 该纳米晶镍镀层因无硫而未观察到异常晶粒生长行为; 同时由于其初始镀态的低能界面结构和一定量退火纳米孪晶的形成, 降低了纳米晶粒生长的驱动力, 导致其热稳定性得到改善, 使得该镀层在 450 °C 退火后, 表现出很小的残余拉应力(约 50 MPa)和较高的硬度(约 HV 400)。

**关键词:** 电沉积; 纳米晶镍; 柔性摩擦; 热稳定性; 晶粒生长

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