

超声条件下脉冲电镀 Ni-纳米 Al_2O_3 复合镀层 及其显微硬度研究

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摘要: 目的 优选脉冲参数, 以获得具有较高显微硬度的复合镀层。方法 超声条件下, 采用脉冲电镀方法制备 Ni-纳米 Al_2O_3 复合镀层。以显微硬度作为性能指标, 对比考察平均电流密度、占空比、频率和施镀时间的影响。结果 Ni-纳米 Al_2O_3 复合镀层的显微硬度随着平均电流密度升高, 占空比增大, 频率升高, 均呈现出先增后减的趋势, 而随着施镀时间延长, 呈现出近似递减的趋势。结论 在平均电流密度 $8 \text{ A}/\text{dm}^2$ 、占空比 0.6、频率 1.5 kHz、施镀时间 3 min 的条件下, 制备的 Ni-纳米 Al_2O_3 复合镀层显微硬度最高, 约为 427.1 HV。较高复含量的纳米微粒有效起到了弥散强化和细晶强化作用, 改善了复合镀层结构致密程度, 进而提高了显微硬度。

关键词: 显微硬度; Ni-纳米 Al_2O_3 复合镀层; 平均电流密度; 占空比; 频率; 施镀时间

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Research on Microhardness of Ni-Nano Al_2O_3 Composite Coatings Prepared by Pulse Electroplating under Ultrasound Condition

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ABSTRACT: Objective To optimize pulse parameters in order to prepare the composite coating with high microhardness. **Methods** Ni-nano Al_2O_3 composite coatings were prepared by pulse electroplating under ultrasound condition, and the influences of average current density, duty cycle, frequency and electroplating time on the microhardness were also investigated. **Results** The microhardness of Ni-nano Al_2O_3 composite coatings increased first and then decreased with the increase of average current density, duty cycle and frequency, and it tended to decrease approximately with the electroplating time prolonging. **Conclusion** The Ni-nano Al_2O_3 composite coating prepared under the condition of average current density $8 \text{ A}/\text{dm}^2$, duty cycle 0.6, frequency 1.5 kHz and electroplating time 3 min had the highest microhardness of about 427.1 HV. It was concluded that nano Al_2O_3 particles of high composite quantity had effects of dispersion strengthening and grain refinement strengthening and improved the density of composite coating structure, thus improving microhardness.

KEY WORDS: microhardness; Ni-nano Al_2O_3 composite coating; average current density; duty cycle; frequency; electroplating time

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在复合电镀过程中引入超声波,能够营造出利好环境条件,有助于复合镀层结构与性能的改善。正是鉴于超声波所彰显出的独特工艺优势,众多学者相继开展了在超声条件下或依托超声环境为载体进行复合电镀的研究。Lecina、司东宏等人在超声条件下分别制备出 Ni-Al₂O₃、Ni-ZrO₂ 纳米复合镀层,均研究了超声波对复合电镀过程及复合镀层结构性能的影响机理^[1-2]。Zheng, Yang 等人在超声条件下分别获得了 Zn-Ni-Al₂O₃、Cu-SiC 纳米复合镀层,并表征了复合镀层的微观结构,分析了复合镀层的硬度、耐腐蚀性能与机械性能^[3-4]。同样在超声条件下,王裕超等^[5]制备出均匀分散着碳纳米管的铜基碳纳米管复合镀层,并初步解释了超声波作用于复合电镀过程的机制。吴蒙华等^[6]结合脉冲电镀工艺,获得了细晶纳米金属陶瓷复合镀层,并对制备工艺进行了优化。李献会等^[7]营造出组合超声环境,进行 Ni-Nd₂O₃ 复合电镀,并探讨了组合超声协同作用对复合镀层耐腐蚀性的影响。

本课题组前期亦开展过超声条件下加载脉冲电流进行复合电镀的研究,得出结论:超声波和脉冲电流作用叠加能明显改善复合镀层的形貌结构,提高性能^[8]。作为后续研究,文中在超声条件下制备 Ni-纳米 Al₂O₃ 复合镀层,并选取显微硬度作为性能指标,系统考察平均电流密度、占空比、频率和施镀时间等主要脉冲参数的影响。

1 试验

1.1 超声脉冲沉积

施镀基材为 $\phi 40\text{ mm} \times 1\text{ mm}$ 的不锈钢板,依照“打磨整平→除油清锈→酸洗活化→冲洗→干燥”的流程进行前处理,以消除非工艺因素可能造成的干扰。阳极采用电解镍板,试验前进行除油清污、冲洗、干燥处理。

采用瓦特型镀镍液作为基础液,组分为:300 g/L NiSO₄ · 6H₂O,50 g/L NiCl₂ · 6H₂O,40 g/L H₃BO₃,15 g/L NaCl,0.1 g/L C₁₂H₂₅SO₄Na。添加 30 g/L 高纯度且经过除杂质和润湿处理的 Al₂O₃ 微粒,其平均粒径约为 70 nm。施加超声波振荡搅拌均匀,制得复合镀 Ni-纳米 Al₂O₃ 的悬浊液。

复合镀在 H6025T 型超声波清洗机的水浴槽中进行。将超声波功率恒定为约 350 W,镀液恒温为 50

℃,仅单独改变脉冲平均电流密度、占空比、频率或施镀时间,制备不同的 Ni-纳米 Al₂O₃ 复合镀层。

1.2 表征及检测

1) 采用 KYKY-2800B 型扫描电镜观察 Ni-纳米 Al₂O₃ 复合镀层的组织结构。

2) 用 HV-1000 型显微维氏硬度计测定复合镀层的显微硬度,加载 0.98 N,保载 10 s。各试样均测试 7 处不同位置,舍去最大、最小值,再取平均值作为测试结果。

3) 采用化学分析法,依照公式 $C = m_{\text{particle}} / m_{\text{coating}} \times 100\%$ (m_{particle} , m_{coating} 分别为微粒、复合镀层的质量)^[9] 计算 Ni-纳米 Al₂O₃ 复合镀层中的微粒复合量。具体流程为:(1)剥离复合镀层,采用 CPA225D 型电子分析天平称取其质量 m_{coating} ;(2)将复合镀层置于硝酸与水体积比约为 1 : 1 的稀硝酸溶液中加热,直至完全溶解;(3)沉淀并过滤,分离出不溶微粒;(4)冲洗、干燥微粒,并称取微粒质量 m_{particle} 。

2 结果与讨论

2.1 平均电流密度对显微硬度的影响

如图 1 所示,随着脉冲平均电流密度升高,显微硬度先增加,后减小,最高为 427.1 HV。平均电流密度低时,较低的阴极过电位限制了晶核的形成速率和生长速度;同时,共沉积过程进行缓慢,被基质金属包覆并嵌埋的纳米微粒量少,起到的强化效果相对弱;这两方面的原因共同导致粗晶、疏松结构的形成(见图 2a),使得显微硬度较低。平均电流密度升高时,强静电吸引和两相界面间的作用力促使更多纳米微粒附着于被镀基底表面,参与共沉积并被包覆。纳米微粒在镀覆层中呈较均匀弥散分布,有效发挥了屏蔽阻碍效果及细晶强化效应,增大了阴极过电位,进而促使结晶细致、结构密实(见图 2b),提高了复合镀层硬度。

平均电流密度也并非越高越有利,过高反倒可能造成负面影响,如图 2c 所示。在高的平均电流密度下,基质金属镀覆过快,虽然微粒被包覆所需的极限时间缩短,但微粒被俘获并嵌埋的速率或难以匹及;并且,可能伴随的析氢副反应对吸附的微粒有一定程度的冲击作用,致使纳米微粒吸附困难,造成复合镀层显微硬度下降^[10-11]。

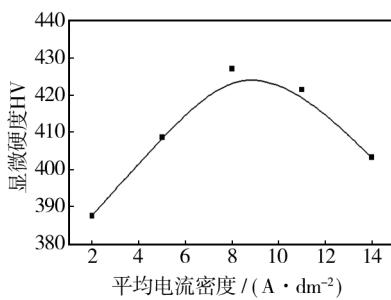


图1 平均电流密度对 Ni-纳米 Al_2O_3 复合镀层显微硬度的影响

Fig. 1 Influence of average current density on the microhardness of Ni-nano Al_2O_3 composite coatings

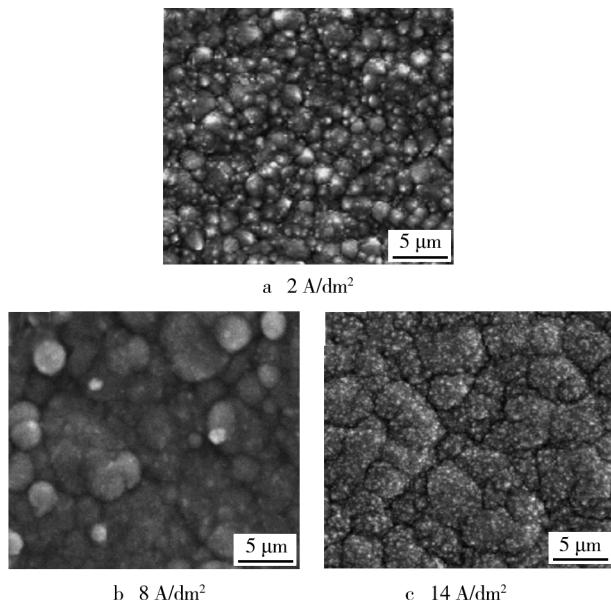


图2 加载不同平均电流密度所得复合镀层的微观结构

Fig. 2 Microstructure of Ni-nano Al_2O_3 composite coatings prepared with different average current densities

2.2 占空比对显微硬度的影响

脉冲占空比与 Ni-纳米 Al_2O_3 复合镀层显微硬度的关系如图 3 所示。与图 1 对比可见,所呈现出的变化趋势与之类似,显微硬度随着占空比增大亦先增后减。分析原因,出现此变化主要归结为占空比改变影响结晶过程和复合镀层中纳米微粒的复合量,进而影响微粒的强化效果以及复合镀层的致密程度。

占空比小时,导通时间短,停歇时间长,停歇期间消耗的离子能够得到有效补充恢复,使得浓度梯度减弱,维系了结晶过程的持续性和稳定性。占空比增大后,镀速加快,微粒被包覆所需的极限时间缩短。另外,高峰值电流密度促使共沉积过程在较大阴极过电

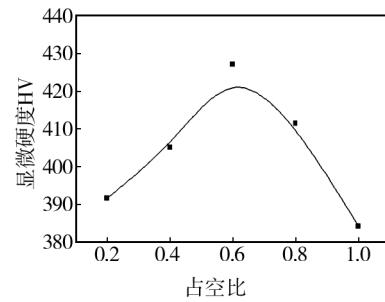


图3 占空比对 Ni-纳米 Al_2O_3 复合镀层显微硬度的影响

Fig. 3 Influence of duty cycle on the microhardness of Ni-nano Al_2O_3 composite coatings

位条件下进行^[12],纳米微粒易附着并嵌埋于复合镀层中,使得复合量增多,如图 4 所示。但占空比增至一定程度,则等同于直流效果,持续进行的共沉积过程可能会导致因扩散传质和对流传质受阻而出现浓差极化,弱化复合镀层结构的同时,也降低纳米微粒的复合量,宏观上表现为显微硬度降低。

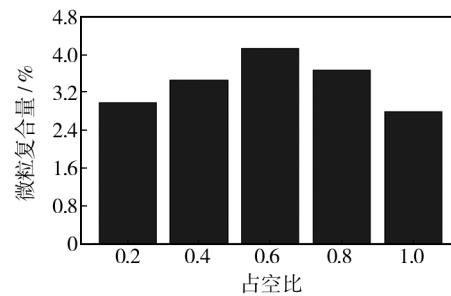


图4 占空比对 Ni-纳米 Al_2O_3 复合镀层中微粒复合量的影响

Fig. 4 Influence of duty cycle on the compound quantity of particles in Ni-nano Al_2O_3 composite coatings

2.3 频率对显微硬度的影响

与脉冲平均电流密度和脉冲占空比的影响规律类似,随着脉冲频率提高,Ni-纳米 Al_2O_3 复合镀层的显微硬度也呈现出先增后减的变化趋势,如图 5 所示。最高硬度值约为 427.1 HV,对应的脉冲频率约为 1.5 kHz。

依据双扩散层(外扩散层和脉动扩散层)模型理论^[13],脉冲频率对被镀基底表面附近的镍离子浓度有着明显的影响,并且与脉冲导通时间存在着一定的关联性。频率低,意味着单位脉冲周期的导通时间短,晶核生长过程受抑制,而形成过程受激励促进,导致结晶紊乱程度加重,位错密度高,引起点阵畸变,形成位错强化和塞积强化。另外,纳米微粒能够冲破双扩散层的阻隔并吸附于被镀基底表面,参与共沉积过程。频率高,则意味着单位脉冲周期的导通时间长,在适宜范畴内有助于加快成核速率,增加成核数目,

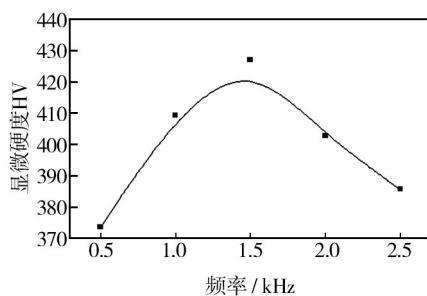
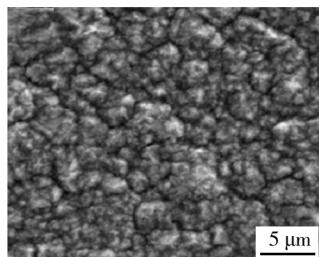
图5 频率对Ni-纳米Al₂O₃复合镀层显微硬度的影响

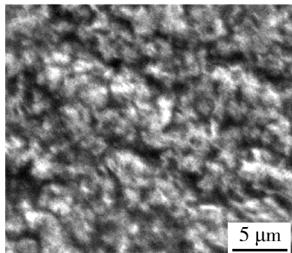
Fig. 5 Influence of frequency on the microhardness of Ni-nano Al₂O₃ composite coatings

改善复合镀层致密程度,进而提高显微硬度。

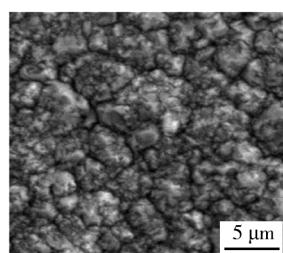
如图6b所示,加载的脉冲频率为1.5 kHz时制备的Ni-纳米Al₂O₃复合镀层结构较为致密,优于其余二者,故其显微硬度相对较高。



a 0.5 kHz



b 1.5 kHz



c 2.5 kHz

图6 加载不同频率制备的复合镀层微观结构

Fig. 6 Microstructure of Ni-nano Al₂O₃ composite coatings prepared with different frequencies

2.4 施镀时间对显微硬度的影响

如图7所示,随着施镀时间延长,Ni-纳米Al₂O₃复合镀层的显微硬度近似递减,这与前面几个工艺参数的影响规律不同。

结合复合电镀机理分析认为,复合电镀过程开始的较短周期内,由于被镀基底表面已粘附一定量的微粒(基底表面微凹坑的弱吸附效应和界面间相互作用力所致^[14]),因而制备的复合镀层虽薄,但其中纳米微粒的复合量仍然相对较高,约为3.72%(如图8所

示),并且复合镀层的结构较致密(见图9a),所以显微硬度较高。随着施镀时间延长,复合镀层随之增厚,尽管其中的纳米微粒复合量增多,但堆叠方式及界面间作用力的影响可能会导致两相间/微粒彼此间相斥,沉积层面排布的微粒密度降低^[15],弥散强化和细晶强化效果不尽理想(见图9b)。这或许是复合镀层显微硬度呈现出递减趋势,而非理论上递增趋势的原因。

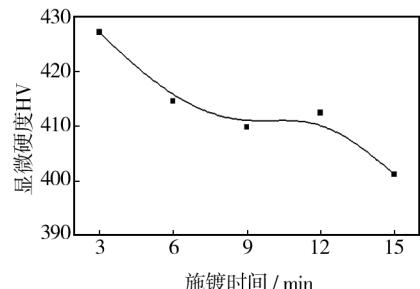
图7 施镀时间对Ni-纳米Al₂O₃复合镀层显微硬度的影响

Fig. 7 Influence of electroplating time on the microhardness of Ni-nano Al₂O₃ composite coatings

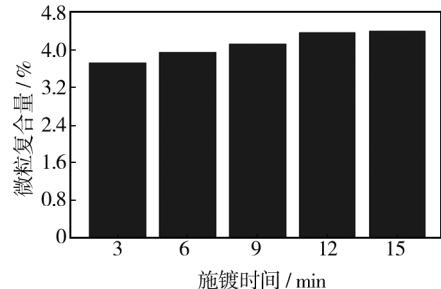
图8 施镀时间对Ni-纳米Al₂O₃复合镀层中微粒复合量的影响

Fig. 8 Influence of electroplating time on the compound quantity of particles in Ni-nano Al₂O₃ composite coatings

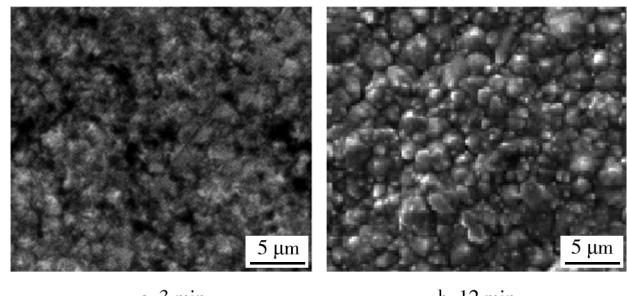
图9 不同施镀时间制备的Ni-纳米Al₂O₃复合镀层微观结构

Fig. 9 Microstructure of Ni-nano Al₂O₃ composite coatings prepared with different electroplating time

3 结论

1) Ni-纳米 Al_2O_3 复合镀层的显微硬度随着平均电流密度升高,占空比增大,频率升高,均呈现出先增后减的趋势,而随着施镀时间延长,呈现出近似递减的趋势。

2) 在平均电流密度8 A/dm²、占空比0.6、频率1.5 kHz、施镀时间3 min的条件下,制备的Ni-纳米 Al_2O_3 复合镀层显微硬度最高,约为427.1 HV。这是因为此时复合镀层中纳米微粒复合量较高,有效起到了弥散强化和细晶强化作用,进而改善了复合镀层结构致密程度。

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