

304 不锈钢双活性屏离子渗氮

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摘要: 目的 考察 304 不锈钢双活性屏离子渗氮技术的可行性及处理效果。方法 利用双活性屏离子渗氮(DASPN)和普通直流离子渗氮(DCPN)两种技术对 304 不锈钢进行低温(420 ℃)硬化处理, 对比分析两种工艺所得渗层的组织, 对比研究两种工艺所得渗层的相结构、硬度和耐蚀性能。结果 采用 DASPN 技术可获得比采用 DCPN 技术更为均匀、致密的渗层, 渗层为单一 S 相层, 硬度为 763HV0.25。电化学测试表明, 两种渗氮技术相比, DASPN 处理获得的渗层耐蚀性能更优。结论 采用 DASPN 技术对 304 不锈钢进行低温硬化处理, 在试样距双屏的距离为 70 mm 时能够获得比 DCPN 更好的渗氮效果。该技术适于工业化推广应用。

关键词: 304 不锈钢; 离子渗氮; 双活性屏; 硬度; 耐蚀性

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Double Active Screen Plasma Nitriding of 304 Stainless Steel

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ABSTRACT: **Objective** To explore the feasibility and thermal effectiveness of double active screen plasma nitriding (DASPN) of 304 stain steel. **Methods** Nitrided layers were prepared on 304 stainless steel substrate by low temperature DASPN technique and common direct-current plasma nitriding (DCPN) technique, respectively. The morphology, phase structure, hardness and corrosion properties of the nitrided layers were comparatively analyzed. **Results** The nitrided layer produced by DASPN technique was more homogenous and compact compared with that produced by DCPN. It was a single S-phase layer and the hardness of the layer was 763HV0.25. Moreover, the layer had a better corrosion resistance than that produced by DCPN. **Conclusion** The thermal effectiveness of 304 steel by DASPN technique was better than that of DCPN treatment, which is probably applicable to commercial process.

KEY WORDS: 304 stainless steel; plasma nitriding; double active screen; hardness; corrosion resistance

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离子渗氮技术能够利用离子的轰击溅射去除不锈钢表面的钝化膜,提高工件表面活性,并且高效、环保,因此广泛应用于奥氏体不锈钢的低温硬化处理^[1-4],但普通直流离子渗氮技术(DCPN)存在打弧、边缘效应及空心阴极效应,导致工件温度不均匀,引起奥氏体不锈钢在渗氮过程中形成Cr的氮化物,从而使其耐蚀性能下降。为解决这一问题,近年来不锈钢活性屏离子渗氮技术(ASPN)成为人们的研究热点^[5-9]。

活性屏离子渗氮是在普通离子炉内增设一个活性屏(铁制圆形笼子),将原本接在工件上的直流负高压接在活性屏上,活性屏发生辉光放电,起到加热工件和提供渗氮载体的作用;工件则罩在活性屏内,并处于浮动电位或施加低的负偏压^[6-7]。活性屏技术的主要优点是改善了离子热处理时炉内温度的均匀性,特别适用于对温度要求严格的不锈钢零件进行低温硬化处理^[7,10]。目前,不锈钢活性屏离子渗氮处理的研究大多采用小尺寸($\phi < 150$ mm)活性屏^[7,11-14],工件处于浮动电位时距活性屏的距离不超过75 mm,否则工件无法渗氮,这致使其工业化应用受到限制。为此,文中采用双活性屏离子渗氮(DASPN)装置对304奥氏体不锈钢进行低温渗氮处理,考察双活性屏离子渗氮处理的可行性及工件渗氮处理后的组织及耐蚀性能。

1 实验

基材为304奥氏体不锈钢,试样尺寸 $\phi 22$ mm \times 5 mm,化学成分(以质量分数计)为:C 0.133%,Si 0.490%,Mn 1.294%,Cr 16.77%,Fe 72.97%,Mo 0.202%,Ni 8.14%。渗氮前,不锈钢试样表面依次用240[#]—1200[#]水磨砂纸磨光,并超声清洗。

渗氮在SLHMC型双重加热多功能离子轰击炉内进行,所用双活性屏装置如图1所示。双活性屏装置置于离子炉工作台上(接负高压),其中外屏尺寸 $\phi 450$ mm,内屏置于外屏中心,尺寸为 $\phi 100$ mm。活性屏材质为不锈钢,网孔尺寸为 $\phi 16$ mm。渗氮试样置于双屏中间,距内、外屏的距离为70 mm。工件与离子炉工作台之间用SiO₂支撑块隔离,工件处于浮动电位。

渗氮开始后,炉内先抽真空至10 Pa左右,然后开启辉光进行升温,并开冷却水。为了与普通离子渗氮效果进行比较,同时安排了304不锈钢的普通离子渗氮。两种工艺采用相同工艺参数:电压550~600 V,

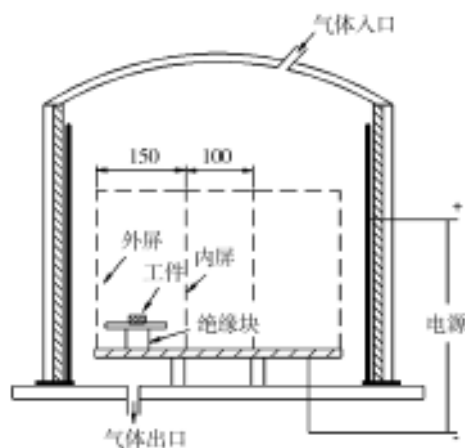


图1 双活性屏离子渗氮示意

Fig. 1 Schematic diagram of DASPN

N₂ 体积分数25%,H₂ 体积分数75%,温度420℃,炉压260~280 Pa,时间5 h。

将渗氮处理后的试样沿横截面切割,并镶样,抛光后,用10 g CuCl₂+50 g HCl+50 g HNO₃ 腐蚀液进行腐蚀。采用蔡司共聚焦显微镜观察试样横截面组织形貌;采用德国布鲁克公司X射线衍射仪(XRD)分析渗层的相组成;采用HVS-1000型显微硬度计测试渗层的表面硬度,硬度取5个测试点的平均值。采用上海辰华CHI660D型电化学测试仪在3.5%(质量分数)NaCl溶液中测试试样处理前后的阳极极化曲线,以此评价耐腐蚀性能,参比电极为饱和甘汞电极,辅助电极为铂片。

2 结果与讨论

2.1 金相分析

图2为304奥氏体不锈钢经过DCPN和DASPN处理后的横截面金相照片。可以看出,两种工艺处理的304不锈钢表面均获得了单一的白亮层,且DASPN处理的白亮层更加均匀致密。DCPN处理的白亮层厚约8.3 μ m,DASPN处理的白亮层厚约5.6 μ m。进行DCPN处理时,工件周围发生辉光放电,通过离子轰击溅射产生大量的活性氮,活性氮由工件表面扩散进入基体而形成白亮层。进行DASPN处理时,工件处于浮动电位,不发生辉光放电,溅射作用很微弱,其氮源由活性屏上溅射下来的携氮粒子提供^[7-8]。由于DCPN工艺离子轰击溅射作用较强,故相同时间内的渗层厚度比DASPN工艺获得的渗层要厚。

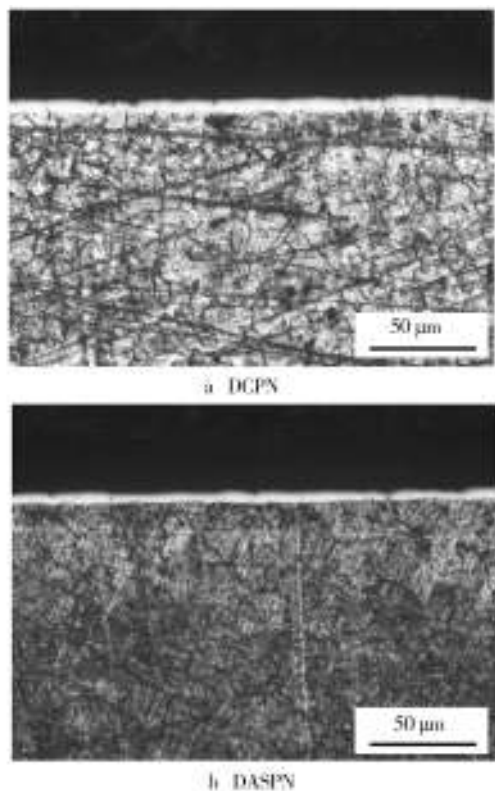


图 2 304 不锈钢离子渗氮后的金相照片

Fig. 2 Optical microstructure of cross-section of 304 stainless steel after DCPN and DASPn treatment at 420 °C

2.2 渗层相分析

图 3 为 304 不锈钢经渗氮处理前后的 XRD 图谱。由于处理温度较低,两种工艺处理获得的渗层主要为氮的扩张奥氏体相。这种相被称为 S 相^[1,3],它是 N 在 Fe 中的过饱和固溶体,具有较高的硬度和较好的耐蚀性能。N 在 γ 中固溶后,原来奥氏体的面心立方晶格(fcc)转变为四方晶格结构(fct),使晶格发生畸变,峰位左移^[7,15],并变宽。

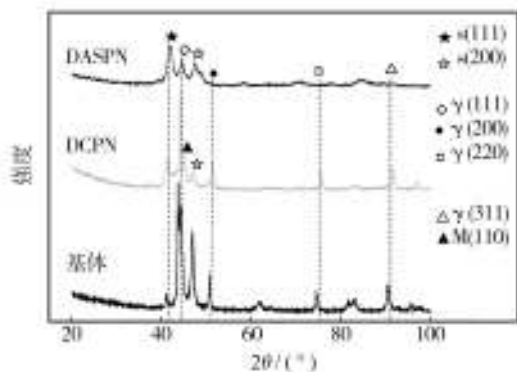


图 3 304 不锈钢及其经离子渗氮处理后的 XRD 图谱

Fig. 3 XRD patterns of the layers before and after treatment by DCPN and DASPn process

2.3 显微硬度

图 4 为 304 不锈钢经渗氮处理前后的表面显微硬度。不锈钢经离子硬化处理后,硬度比基体有显著提高,DCPN 工艺处理的渗层硬度略高于 DASPn 工艺。分析认为,在 DCPN 处理过程中,由于边缘效应的存在使工件温度不均匀,虽然温度表显示 420 °C,但工件处于辉光放电状态,实际温度与测量温度存在偏差,工件局部温度过高则会生成少量 Cr 的氮化物,使渗层硬度提高;而在 DASPn 处理过程中,由于工件不产生辉光放电,工件温度均匀,渗层为单一 S 相层,未生成 Cr 的氮化物。不锈钢经低温渗氮后硬度提高主要是由于固溶强化,渗层中未生成大量 Cr 的氮化物,因此硬度比高温渗氮时要低。

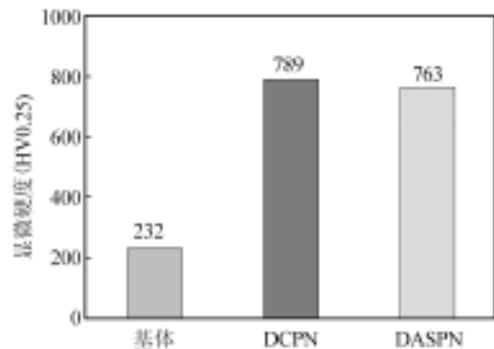


图 4 304 不锈钢及其经离子渗氮处理后的表面硬度

Fig. 4 Surface hardness of treated 304 stainless steel by DCPN and DASPn process

2.4 电化学分析

图 5 为 304 不锈钢经渗氮处理前后的阳极极化曲线。可以看出,两种渗氮试样的自腐蚀电位高于基体,说明其表面耐蚀性能优于基体。同时,DASPn 处

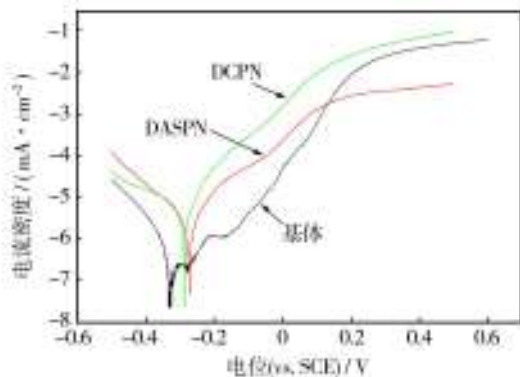


图 5 304 不锈钢及其经离子渗氮处理后的阳极极化曲线

Fig. 5 Polarization curves of samples before and after DCPN and DASPn treatment

理的试样自腐蚀电位高于 DCPN 处理的试样,说明 304 不锈钢经过低温 DASPEN 处理后获得了更优异的耐蚀性能。

3 讨论

活性屏离子渗氮处理时,当工件处于浮动电位,距离活性屏较远的试样渗氮效果较差或者渗不上氮,因此已发表的文献中大多采用小尺寸活性屏($\phi < 150$ mm),由于尺寸较小,工业化应用受到限制。本研究中采用双屏结构,比单屏体积扩大了约 20 倍,大大提高了装炉空间。另外,离子渗氮过程中,两个活性屏均发生辉光放电,从活性屏上溅射下来的活性氮的载体数量也大大提高,改善了单屏渗氮时的渗层质量。结合图 2 的渗层形貌和图 3 的 XRD 图谱可知,304 不锈钢经过 DASPEN 处理,可获得较理想的渗氮效果。

不锈钢离子渗氮对温度要求严格,DASPEN 处理保证了炉内试样温度的均匀性,能够得到单一的 S 相层,有利于其耐蚀性能的提高。DCPN 处理虽然是在低温(420 ℃)下进行,但由于温度均匀性差,会导致少量 Cr 的氮化物形成(由于量少,XRD 未检测出),这从图 2a 的金相形貌可以看出,渗层内晶界有被腐蚀的现象。

奥氏体不锈钢在 DCPN 处理时,表面的钝化膜经离子轰击很容易被去除;在 DASPEN 处理时,虽然试样处于浮动电位,表面未发生辉光放电,但实验证明仍能进行渗氮处理。这说明在双活性屏空间内,存在随气流穿过活性屏、撞击到试样表面的活性粒子(活性屏上溅射下来的粒子、活性碳原子和氢原子)以及在电场作用下向阳极运动的电子。虽然它们的轰击溅射作用比正离子直接轰击要弱得多,但足以破坏不锈钢表面的钝化膜,而且在低氧富氢的还原气氛中,无法重新生成新的钝化膜,从而使奥氏体不锈钢活性屏渗氮过程得以顺利进行。

4 结论

1) 采用双活性屏装置对 304 奥氏体不锈钢进行渗氮处理,当试样距双屏之间的距离为 70 mm 时,亦能获得较好的渗氮效果。当试样处于浮动电位时,采用双屏结构进行渗氮处理可有效扩大装炉体积,有利于 304 不锈钢活性屏离子渗氮处理的工业化应用。

2) 在文中的工艺条件下,304 不锈钢经双活性屏

渗氮获得的渗层相比普通直流离子渗氮获得的渗层,组织更加致密,为单一 S 相层,具有优异的耐蚀性能。

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(下转第 115 页)

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