

引用格式: 黄居峰, 吕乃欣, 徐秀清, 等. 两种焊后热处理工艺对 Incoloy 825 镍基合金氩弧焊接头微观组织与力学性能的影响[J]. 材料工程, 2026, 54(3): 122-130.

HUANG Jufeng, LYU Naixin, XU Xiuqing, et al. Effect of two post-weld heat treatment on microstructure and mechanical properties of Incoloy 825 nickel base alloy argon arc welded joint[J]. Journal of Materials Engineering, 2026, 54(3): 122-130.

两种焊后热处理工艺对 Incoloy 825 镍基合金氩弧焊接头微观组织与力学性能的影响

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摘要: 针对镍基合金在焊接后焊缝中产生微观组织偏析, 导致接头力学性能下降等问题。对比研究了两种焊后热处理工艺对 Incoloy 825 镍基合金氩弧焊接头微观组织与力学性能的影响。组织分析表明: 经两种工艺热处理后, 母材 (base material, BM) 中原细小的晶界碳化物明显粗化, 焊缝区 (weld zone, WZ) 枝晶间析出大量 δ 和 Laves 相。固溶+双级时效热处理可诱导 BM 晶内析出 γ' 相, 且导致未混合区 (unmixed zone, UMZ) 与热影响区 (heat-affected zone, HAZ) 界面产生微裂纹, HAZ 形成微孔洞。力学性能测试分析表明: 经两种工艺热处理后, WZ 显微硬度明显增加。其中, 固溶+双级时效热处理 HAZ 和 BM 硬度达到接头最高值。经固溶+单级时效热处理接头抗拉强度略有下降, 而均匀伸长率提高了 9.2%, 拉伸断裂位于 BM 区。经固溶+双级时效热处理接头抗拉强度显著提高, 达 795 MPa, 提高了 17.3%, 而均匀伸长率有所下降, 拉伸断裂位于 UMZ/HAZ 界面及部分 HAZ, 拉伸断口呈韧脆混合断裂特征。

关键词: 镍基合金; 氩弧焊; 焊后热处理; 微观组织; 力学性能

doi: 10.11868/j.issn.1001-4381.2025.000087 CSTR: 32421.14.j.issn.1001-4381.2025.000087

中图分类号: TG444 文献标识码: A 文章编号: 1001-4381(2026)03-0122-09

Effect of two post-weld heat treatment on microstructure and mechanical properties of Incoloy 825 nickel base alloy argon arc welded joint

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Abstract: To tackle the issue of microstructural segregation in nickel-based alloy weldments and the consequent degradation of joint mechanical properties, this study delves into the impacts of two post-weld heat treatment schedules on the microstructural features and mechanical behavior of gas tungsten arc-welded Incoloy 825 nickel-based alloy joints. The results reveal that both heat treatment procedures lead to a notable coarsening of fine grain boundary carbides in the base metal (BM). Moreover, the γ' phase can be precipitated within BM grains through solution treatment combined with two-stage aging. Both post-weld heat treatments facilitate extensive precipitation of the δ and Laves phases in the WZ interdendritic zones, effectively alleviating segregation. Nevertheless, joints subjected to solution treatment and two-stage aging develop microcracks at the interface between the unmixed zone (UMZ) and the heat-affected zone (HAZ), along with the formation of micropores within the HAZ. Mechanical characterization indicates a substantial increase in microhardness in the weld zone (WZ) after both heat treatment protocols. Solution treatment with single-stage aging maintains hardness levels in the HAZ and BM that are comparable to those in the as-welded state. Joints treated with solution treatment and single-stage aging

show a marginal reduction in tensile strength, accompanied by a 9.2% improvement in uniform elongation, and fracture exclusively occurs within the BM. Conversely, the tensile strength of joints treated with solution treatment and two-stage aging significantly rises to 795 MPa, an increase of 17.3%, while the uniform elongation decreases. The fracture localization shifts to the UMZ/HAZ interfacial regions and adjacent HAZ areas, presenting a ductile-brittle mixed fracture morphology.

Key words: nickel-based alloy; argon arc welding; post-weld heat treatment; microstructure; mechanical property

Incoloy 825 镍基合金以 Ni-Fe-Cr 为基体, 由于其具有优异的力学性能和耐高温腐蚀性能, 被广泛应用于航空航天、石油石化和核能工业中^[1-4]。镍基合金部件通常采用电子束焊^[1]、激光焊^[5]和钨极惰性气体保护焊 (TIG)^[6]焊接。其中, TIG 设备价格低廉, 操作灵活方便, 焊接成本低, 并且适合全位置焊接, 成为 Incoloy 825 镍基合金管焊接的首选技术之一。通常, 镍基合金焊后容易在焊缝金属中产生微观组织偏析和残余应力等, 这会对焊接接头力学性能产生不利影响^[7-9]。焊后热处理作为一种补救措施应用于镍基合金焊接接头中, 通过改变焊态组织和减轻焊接残余应力, 以获得最佳的焊接接头力学性能。

Xie 等^[10]在 K4750 镍基合金 TIG 焊接头微观组织和力学性能的研究中发现, 固溶+时效热处理产生的 50 nm 球状 γ' 相, 使焊缝区显微硬度从 250HV 提高到 380HV, 并且接头拉伸强度提高了 1.9%, 但伸长率下降了 81.5%。Taheri 等^[11]在时效热处理对激光焊接 GTD-111 镍基合金接头微观组织和力学性能影响的研究中发现, 采用 845 °C/24 h 空冷时效处理工艺, 热影响区析出较粗大的 γ' 相, 导致热影响区产生应变时

效裂纹, 接头拉伸性能显著下降, 拉伸断裂位于热影响区。Ramkumar 等^[12]在时效热处理对 X750 镍基合金 A-TIG 焊接头微观组织和力学性能的影响研究中发现, 采用 705 °C/20 h 空冷时效处理工艺, γ' 析出相较细小, 接头拉伸性能得到提升, 但拉伸断裂位于焊缝区。

综上所述, 目前的焊后热处理工艺对改善镍基合金焊态微观组织和提高接头力学性能并不理想, 并且针对 Incoloy 825 镍基合金氩弧焊接头焊后热处理的研究鲜有报道。因此, 本工作采用两种热处理工艺对 Incoloy 825 镍基合金氩弧焊接头进行焊后热处理, 对比研究两种热处理工艺对焊接接头微观组织与力学性能的影响, 旨在制定 Incoloy 825 镍基合金焊后热处理工艺提供参考。

1 实验材料与方法

1.1 实验材料

实验母材为退火态 Incoloy 825 镍基合金管, 规格为 $\Phi 114 \text{ mm} \times 5 \text{ mm}$; 焊材选用直径为 2.4 mm 的 ER-NiCrMo-3 焊丝。母材及焊材化学成分见表 1。

表 1 母材及焊材化学成分(质量分数/%)

Table 1 Chemical compositions of base material and welding material (mass fraction/%)

| Material | C | Cr | Ni | Si | Mn | Ti | P | S | Nb | Mo | Cu | Fe |
|------------------|-------|------|------|------|------|------|-------|-------|-----|-----|-----|------|
| Base material | 0.011 | 22.4 | 39.1 | 0.11 | 0.50 | 0.95 | 0.016 | 0.001 | — | 3.0 | 2.0 | Bal. |
| Welding material | 0.02 | 22.0 | 64.0 | 0.14 | 0.20 | — | — | — | 3.5 | 9.0 | — | 1.0 |

1.2 实验方法

母材两管端加工成 V 型坡口, 单边坡口角度为 $(30^\circ \pm 2^\circ)$, 对坡口 20 mm 范围管内外进行清理并露出金属光泽。清理完成后, 将两管端固定在专用工作台上进行组对, 组对间隙为 3.0~3.5 mm。焊机选用 OTC VRTP400 型焊接电源, 焊接工艺参数见表 2。焊后试样热处理在 BDL-1 真空管式炉中进行。两种焊后热处理工艺分别为 980 °C \times 1 h(炉内充氩快冷至室温)+780 °C \times 8 h(空冷)、980 °C \times 1 h(炉内充氩快冷至室温)+780 °C \times 8 h(炉冷至 650 °C)+650 °C \times 8 h

(空冷), 即固溶+单级和固溶+双级时效处理。

采用线切割沿焊缝横截面切取金相试样, 经磨制、抛光后, 采用体积比 $\text{HNO}_3:\text{HCl}=3:1$ 的王水溶液进行侵蚀。采用 Axion Vert. A1 型光学显微镜进行金相组织观察。采用 ZEISS Merlin compact 型场发射扫描电子显微镜 (SEM) 进行微观组织和拉伸断口形貌观察, 采用 SEM 携带的能谱分析仪 (EDS) 对微观组织进行点扫和面扫, 测定其元素含量和分布。采用 HXD-1000TMC 型显微硬度计对接头横截面显微硬度进行测试, 载荷为 2.94 N, 保压时间为 10 s。采用

表2 焊接工艺参数

Table 2 Welding process parameters

| Welding layer | Welding current/A | Welding speed/ (mm·min ⁻¹) | Types and polarity of current | Front-side shielding Ar gas flow/(L·min ⁻¹) | Back-side purging Ar gas flow/(L·min ⁻¹) |
|---------------|-------------------|---|----------------------------------|--|---|
| Root pass | 75-85 | 35-40 | Straight polarity direct current | 10-15 | 5-6 |
| Filler pass | 90-95 | 45-50 | Straight polarity direct current | 10-15 | — |
| Cover pass | 90-95 | 45-50 | Straight polarity direct current | 10-15 | — |

Instron 8801型试验机进行室温拉伸试验,拉伸实验执行GB/T228—2002标准,拉伸试样平行段尺寸为25 mm×20 mm×5 mm,拉伸速率为5 mm·min⁻¹。采用XRD-6000型X射线衍射仪(XRD)对接头物相进行分析。

2 结果与分析

2.1 接头组织

2.1.1 宏观组织

图1为焊接接头宏观组织形貌。焊缝呈碗状,成形良好,未见明显焊接缺陷。焊接接头分为四个区,分别为母材区(base material, BM)、焊缝区(weld zone, WZ)、未混合区(unmixed zone, UMZ)和热影响区(heat-affected zone, HAZ)。

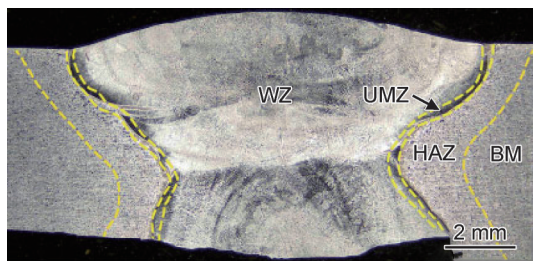


图1 样品的微观形貌

Fig.1 Micromorphology of samples

2.1.2 BM及其热处理微观组织

图2(a)为BM热处理前SEM微观组织,其组织主要为奥氏体,奥氏体晶粒内存在较多退火孪晶,并且沿奥氏体晶界有细小的白色第二相析出。由EDS点扫结果可知(图2(b)),点1的白色第二相富Cr和富Mo,与点1相比较,点2的奥氏体基体Cr和Mo含量较少。Xu等^[13]对第二相进行了表征,表明第二相为M₂₃C₆或M₆C碳化物。并且认为随着Mo和Cr元素在碳化物中相对含量的变化,两种碳化物可相互转变。M₂₃C₆随着Mo元素含量的增加可转变为M₆C。相反,随着Cr元素含量的增加可转变为M₂₃C₆。相互转变描述如式(1)所示:



图2(c),(d)分别为BM经固溶+单级时效和固溶+双级时效热处理工艺后的SEM微观组织。与热处理前BM相比较,BM经两种热处理工艺后晶界析出相明显粗化,在晶界呈不连续网状分布。固溶+双级时效热处理比单级时效的晶界析出相更多。Tan等^[14]研究表明,晶界第二相颗粒的存在可防止晶界迁移,但第二相颗粒沿晶界呈网状分布时,对接头延展性产生不利影响。由图2(c)放大区域EDS面扫结果可知,析出相富Cr、Mo和C,表明析出相为M₂₃C₆或M₆C碳化物。此外,与固溶+单级时效热处理相比较(图2(c)),固溶+双级时效热处理BM晶内析出大量细小γ'-(Ni₃Al, Ti)相(图2(d)黄色箭头所示)。表明固溶+双级时效热处理可诱导γ'相析出。

2.1.3 热处理前后WZ微观组织

图3(a)为热处理前WZ的SEM微观组织,主要为柱状和胞状枝晶以及枝晶间组织。由WZ的EDS面扫结果可知(图3(b)),枝晶间组织主要富Mo和富Nb,其次富Ti和C,而Cr和Ni相未见明显变化。研究表明,WZ富Mo和富Nb相归因于焊缝金属凝固过程中Mo和Nb的偏析^[14-15]。此外,根据Silva等^[16]的研究结果可知,第二相为复杂的碳化物颗粒(例如Nb₂C、(NbTi)C)和Laves相。

图3(c),3(d)分别为焊后接头经固溶+单级和双级时效热处理后WZ的SEM微观组织。枝晶间均析出大量针状和不规则块状组织,由图3(c)放大区域EDS面扫结果可知,析出相富Mo和Nb,贫Cr和Ni(图3(e))。大量研究表明,热处理后形成的针状和块状析出相分别为δ相-Ni₃(Nb, Mo)和Laves相-(Ni, Cr, Fe)₂(Nb, Mo, Ti)^[17-20]。与固溶+单级时效热处理相比较(图3(c)),固溶+双级时效热处理δ析出相有所减少,分布较稀疏。同时可见枝晶间出现了明显缝隙(图3(d))。枝晶间缝隙的形成应与焊后时效处理焊缝应力松弛密切相关^[21]。图3(f)为热处理前后WZ的XRD图谱。从图中可见,热处理前WZ组织中存在NbC和Laves相衍射峰;固溶+单级时效热处理后,δ相衍射峰最强,而固溶+双级时效热处理后,δ相衍射峰强度明显减弱,这说明固溶+双级时效热处理后δ

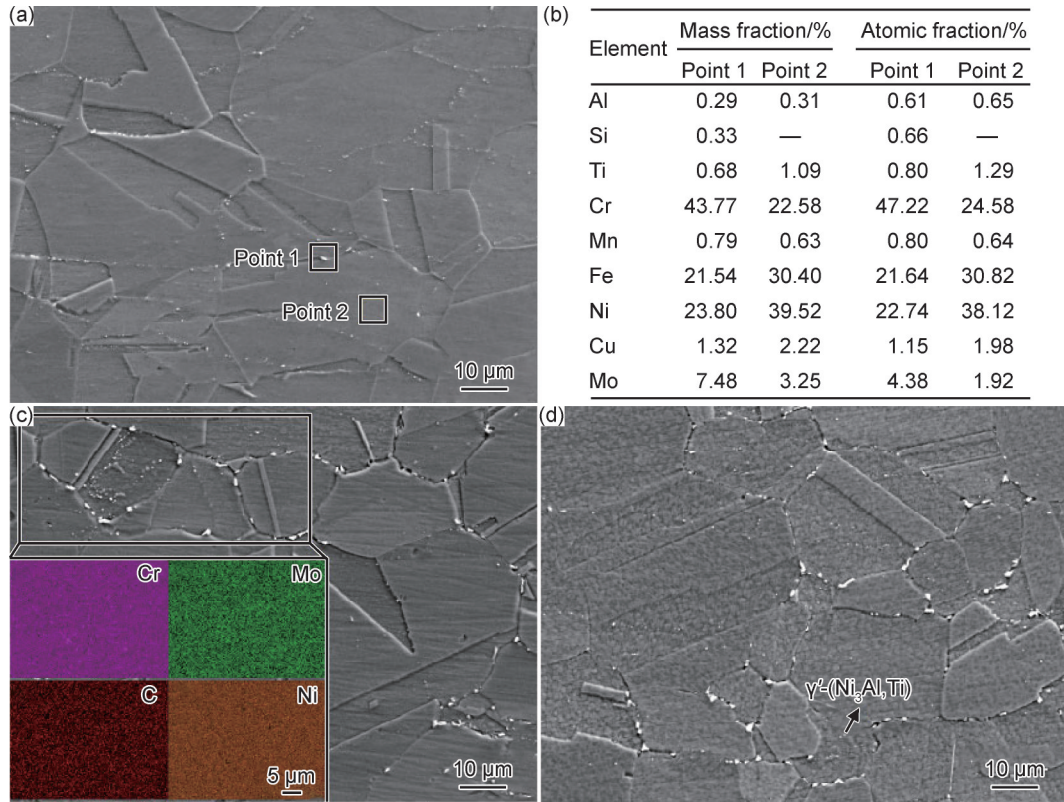


图2 BM热处理前后SEM微观组织

(a)BM热处理前;(b)BM热处理前EDS点扫结果;(c)固溶+单级时效热处理;(d)固溶+双级时效热处理

Fig.2 SEM microstructure before and after BM heat treatment

(a)before BM heat treatment;(b)EDS point scan results before BM heat treatment;(c)solution treatment with single-stage aging heat treatment;(d)solution treatment with two-stage aging heat treatment

析出相明显减少。这些与微观组织分析结果相一致。

2.1.4 热处理前后UMZ及UMZ/HAZ界面微观组织

图4(a)为热处理前UMZ及UMZ/HAZ界面SEM微观组织。UMZ的形成是由于填充材料与BM熔点和化学成分不同,在焊接过程中仅有少部分BM熔化,并且在凝固阶段未被填充材料稀释而形成的分流层^[22-23]。由EDS点扫结果可知(图4(b)),点1~4的UMZ最接近BM的化学成分(质量分数),表明UMZ为部分BM熔化而形成。此外,在UMZ/HAZ界面可见平行于熔合界面的部分重熔和再凝固的液化晶界(图4(a),(c)中白色箭头所示),Moosavy等^[24]研究表明,这些位置(即液化晶界)是液化裂纹成核和扩展的适宜场所。由图4(c)可见,固溶+单级时效热处理后,UMZ/HAZ界面液化晶界处有白色析出相形成,其与BM析出相一致,为 $M_{23}C_6$ 或 M_6C 碳化物。与固溶+单级时效热处理相比较,固溶+双级时效热处理后UMZ/HAZ界面液化晶界处产生明显的晶间微裂纹(图4(d)黄色箭头所示)。David等^[25]研究表明,镍基合金焊后时效热处理过程中,晶间开裂敏感性取决

于几个因素,包括成分、晶粒尺寸、焊接残余应力和焊后热处理温度与时间。根据本工作中固溶+单级时效热处理后,UMZ/HAZ界面液化晶界处未发生开裂的事实,推断固溶+双级时效热处理后,两个区界面液化晶界出现的开裂为焊后热处理过程中产生。裂纹的产生归因于三方面的原因:一是较长时间时效处理导致两个区界面焊接热应力松弛^[25];二是液化晶界析出碳化物的脆化作用;三是液化晶界较弱的固-液结合力^[26]。

2.1.5 热处理前后HAZ微观组织

图5为热处理前后HAZ微观组织。由图5(a)可见,热处理前HAZ中未见原BM晶界碳化物,并且未见晶界开裂等缺陷。由图中的金相组织可见,与BM相比较,HAZ晶粒发生了粗化。由图5(b)固溶+单级时效热处理后HAZ微观组织可见,沿晶界有较多析出相形成,但未见晶界有开裂现象。由图5(b)放大区域面扫结果可知(图5(c)),析出相富含Cr、Mo和C,表明固溶+单级时效热处理后HAZ析出相为 $M_{23}C_6$ 或 M_6C 碳化物。与固溶+单级时效热处理相比较,固溶+双级时效热处理后HAZ沿晶界有少量微孔洞形

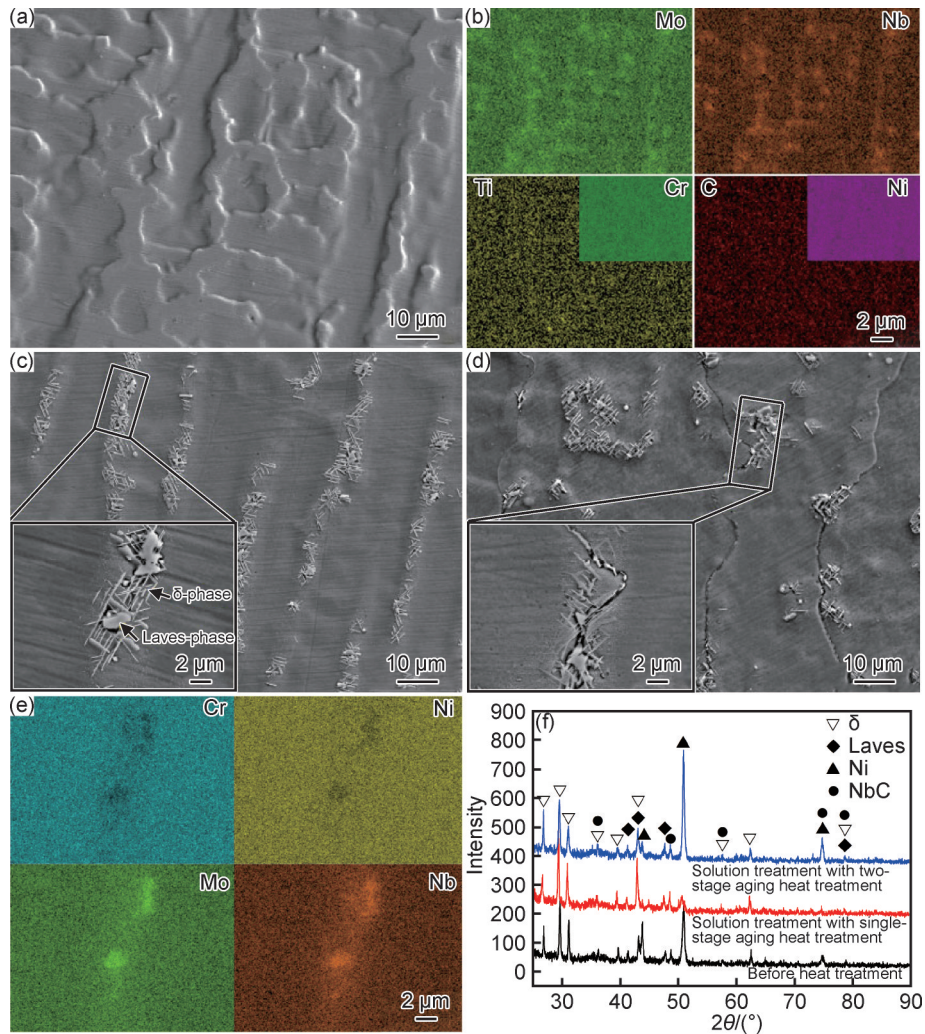


图3 热处理前后WZ微观组织

(a)WZ热处理前;(b)WZ热处理前EDS面扫结果;(c)固溶+单级时效热处理;(d)固溶+双级时效热处理;
(e)固溶+单级时效热处理WZ放大区域EDS面扫结果;(f)热处理前后WZ XRD图谱

Fig.3 Microstructure of WZ before and after heat treatment

(a)before WZ heat treatment;(b)EDS scanning results before WZ heat treatment;(c)solution treatment with single-stage aging heat treatment;
(d)solution treatment with two-stage aging heat treatment;(e)EDS surface scan results of solution treatment with single-stage aging
WZ amplification area;(f)XRD pattern of WZ before and after heat treatment

成(图5(d)放大区域黄色箭头所示)。这些表明双级时效热处理过程中,因时效时间过长而形成的较多 $M_{23}C_6$ 或 M_6C 碳化物脆性组织对HAZ有不利影响。

2.2 接头力学性能

2.2.1 热处理前后接头显微硬度

热处理前后接头显微硬度如图6所示。由于UMZ较窄,显微硬度图中未标注。与热处理前BM相比较,热处理前接头WZ显微硬度明显增加,平均硬度值为193HV,而HAZ发生了软化现象(虚线椭圆所示)。WZ显微硬度增加是由于枝晶间固溶的富Mo和富Nb第二相所致^[4],HAZ软化是焊后晶粒粗化造成的。经固溶+单级时效热处理后,WZ硬度进一步增加,平均硬度值为216HV,而HAZ和BM硬度变化不

明显。WZ硬度的进一步增加是由于热处理后微观组织中析出了较多 δ 和Laves相的缘故。固溶+双级时效热处理后,WZ硬度值与固溶+单级时效热处理接近。然而,其HAZ和BM的硬度显著增加(实线椭圆所示),最高硬度值分别达到254HV和243HV,为热处理前接头HAZ和BM最高硬度的1.3倍和1.4倍。HAZ硬度的增加与固溶+双级时效热处理后,UMZ/HAZ和HAZ形成的碳化物硬化组织有关;BM硬度增加归因于形成的大量细小 γ' 硬化相。

2.2.2 BM及热处理前后接头拉伸性能

图7为BM及热处理前后接头拉伸性能,拉伸性能测试结果及标准值见表3。由图7(a)应力-应变曲线可见,热处理前焊接接头抗拉强度相比BM略有提

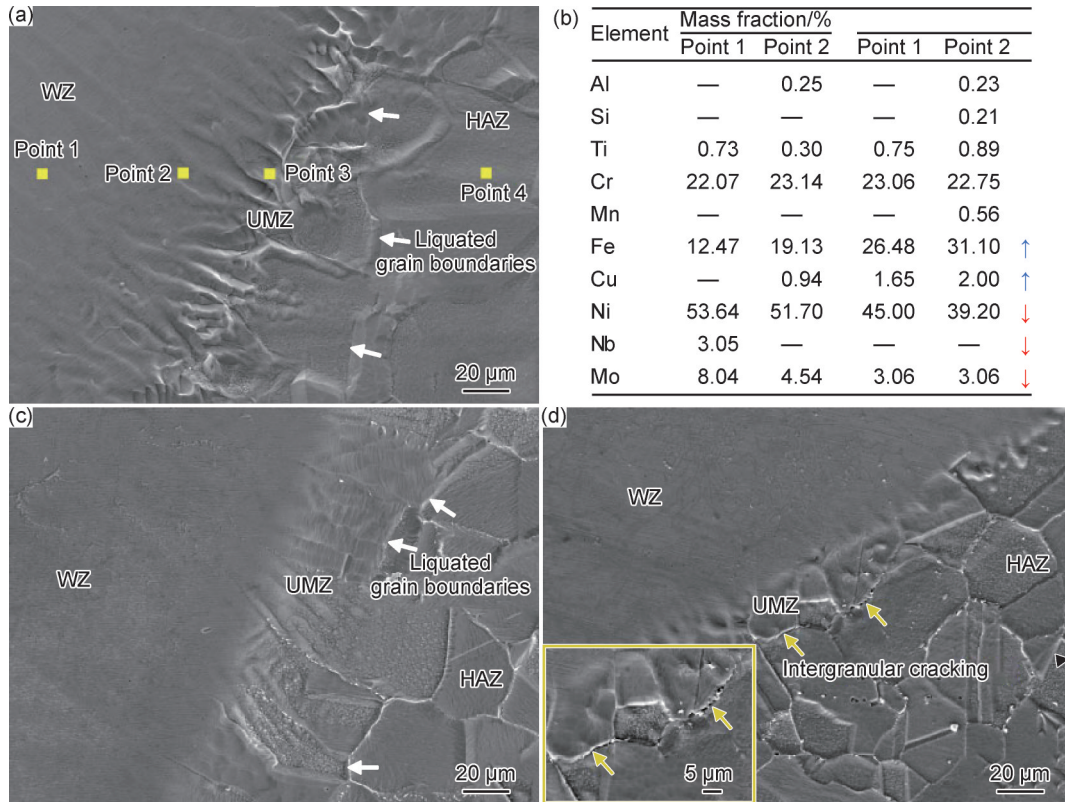


图 4 热处理前后 UMZ 及 UMZ/HAZ 界面组织

(a) 热处理前; (b) 热处理前 EDS 点扫结果; (c) 固溶+单级时效热处理; (d) 固溶+双级时效热处理

Fig.4 UMZ and UMZ/HAZ interface structures before and after heat treatment

(a) before heat treatment; (b) EDS point scan results before heat treatment; (c) solution treatment with single-stage aging heat treatment; (d) solution treatment with two-stage aging heat treatment

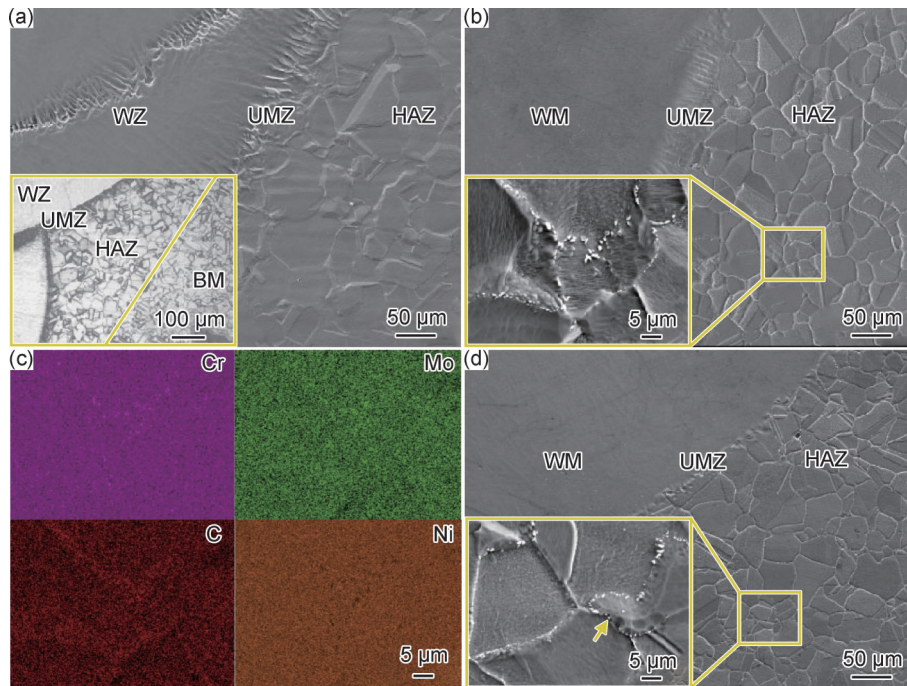


图 5 热处理前后 HAZ 微观组织

(a) HAZ 热处理前; (b) 固溶+单级时效热处理; (c) 固溶+单级时效热处理 EDS 面扫结果; (d) 固溶+双级时效热处理

Fig.5 HAZ microstructure before and after heat treatment

(a) before HAZ heat treatment; (b) solution treatment with single-stage aging heat treatment; (c) EDS surface scan results of solution treatment with single-stage aging heat treatment; (d) solution treatment with two-stage aging heat treatment

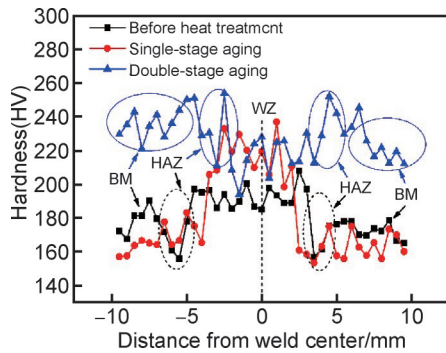


图6 热处理前后焊接接头显微硬度

Fig.6 Microhardness of welded joint before and after heat treatment

高,但均匀伸长率下降了11.6%,这应与焊后接头WZ硬度增加有关。与热处理前接头相比较,经固溶+单级时效热处理后,接头抗拉强度有所下降,但均匀伸长率提高了9.2%,拉伸断裂位于BM区。然而,经固

溶+双级时效热处理后,接头抗拉强度显著提高,达795 MPa,提高了17.3%。而均匀伸长率为37.0%,有所下降。拉伸断裂位于UMZ/HAZ界面和部分HAZ(图7(b),(c),(d))。接头抗拉强度的提高归因于固溶+双级时效热处理后接头微观组织中 δ 、Laves和 γ' 硬化相析出的缘故。研究表明,这些析出相与基体之间存在共格或半共格关系,能够有效阻碍拉伸时的位错运动,从而使得接头强度提高^[11]。值得注意的是,固溶+双级时效热处理接头拉伸过程中出现了瞬时开裂现象(图7(a)中拉伸曲线放大段所示)。该现象与固溶+双级时效热处理时UMZ/HAZ界面产生的晶间微裂纹(图4(d)),以及HAZ形成的微孔洞(图5(d)和图7(d))有关。固溶+双级时效热处理接头拉伸断口形貌如图7(d)中左上图可见,断口中存在韧窝、脆性台和沿晶裂纹,呈韧脆混合断裂特征。综上

表3 母材及热处理前后接头拉伸性能测试结果及标准值

Table 3 Test results and standard values of tensile properties of base metal and joints before and after heat treatment

| Type | Tensile strength/MPa | Percentage uniform elongation/% | Standard values of tensile strength/MPa |
|--|----------------------|---------------------------------|---|
| Base material | 672±5 | 43.0±0.17 | 665 |
| Pre-heat treatment joint | 698±10 | 38.0±0.8 | 585 |
| Solution treatment with single-stage aging joint | 686±8 | 41.5±0.5 | 585 |
| Solution treatment with two-stage aging joint | 795±12 | 37.0±1.2 | 585 |

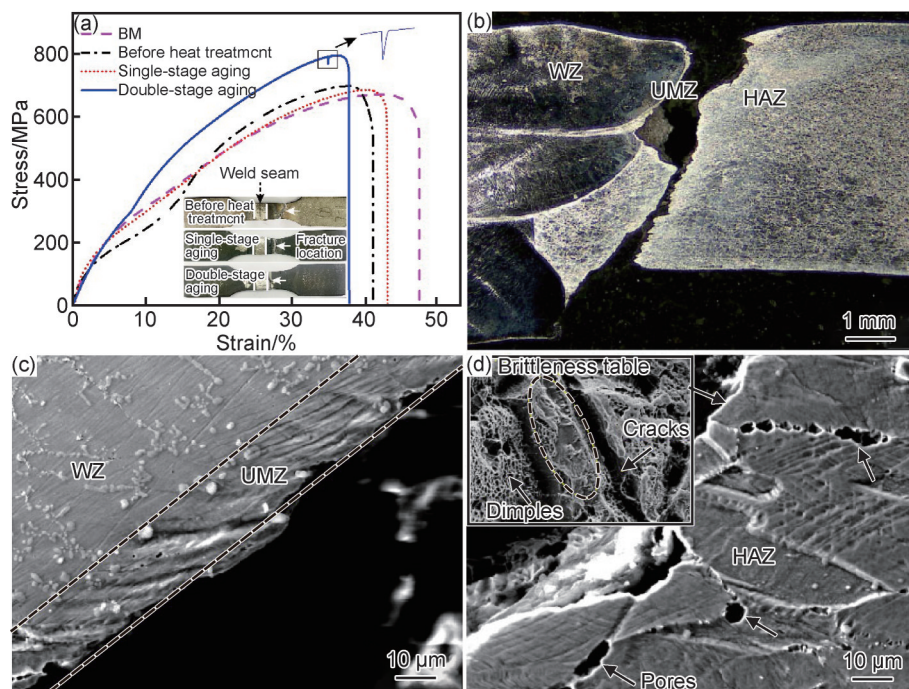


图7 BM及热处理前后焊接接头拉伸性能

(a)应力-应变曲线;(b)固溶+双级时效热处理接头断裂宏观形貌;(c)图b中UMZ放大图;(d)图b中HAZ放大图及断口形貌

Fig.7 Tensile properties of BM and welded joints before and after heat treatment

(a)stress-strain curves;(b)fracture macro morphology of solution treatment with two-stage aging joints;

(c)magnification of UMZ in Fig.(b);(d)HAZ enlargement and fracture morphology in Fig.(b)

可知, Incoloy 825 镍基合金焊接接头经两种工艺热处理后, 其接头力学性能均得到明显改善, 符合 JB/T 4756—2006《镍及镍合金制压力容器》对镍基合金接头的要求。

3 结论

(1) 经两种工艺热处理后, BM 晶界 $M_{23}C_6$ 或 M_6C 碳化物明显粗化, 并且固溶+双级时效热处理可诱导 BM 晶内析出 γ' 相。热处理前接头 WZ 枝晶间组织富 Mo 和富 Nb, 微观组织偏析严重。经两种工艺热处理后, WZ 枝晶间析出大量 δ 和 Laves 相, 微观组织偏析得到缓解。经固溶+双级时效热处理后, UMZ/HAZ 界面产生了微裂纹, HAZ 形成了微孔洞。

(2) 经两种工艺热处理后, WZ 显微硬度明显增加, 平均硬度值为 216HV, 并且固溶+单级时效热处理, HAZ 和 BM 硬度变化不明显。而固溶+双级时效热处理, HAZ 和 BM 硬度显著增加, 最高硬度值分别达到 254HV 和 243HV, 为热处理前接头对应区域最高硬度值的 1.3 和 1.4 倍。

(3) 经固溶+单级时效热处理接头抗拉强度略有下降, 而均匀伸长率提高了 9.2%, 拉伸断裂位于 BM 区。经固溶+双级时效热处理接头抗拉强度显著提高, 达 795 MPa, 提高了 17.3%。而均匀伸长率有所下降。拉伸断裂位于 UMZ/HAZ 界面和部分 HAZ, 断口呈韧脆混合断裂特征。

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基金项目: 中国石油天然气股份有限公司炼油化工和新材料分公司科学研究与技术开发项目(2024LH-02)

收稿日期: 2025-02-18; **录用日期:** 2025-04-18

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