

镍基焊接材料熔滴特性及焊态熔敷金属与低温钢力学性能匹配

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摘要: 以真空冶炼 + 电渣重熔二步法制取镍合金焊芯, 设计高碱度药皮形成复层梯度型熔渣, 并研制出 ENiCrMo-6 深冷镍合金专用焊条. 对研制焊条熔敷金属和采用 ENiCrMo-6 型异质材料焊接 9Ni 钢接头的力学性能进行试验. 借助 Acuteye 高速摄像技术研究熔滴过渡及熔池流动特性, 采用 JSM-6360LV 型扫描电子显微镜分析复层熔渣结构. 对焊缝和热影响区进行观察和分析以从不同角度探讨低温断裂行为. 结果表明, 针对液态镍合金熔滴过渡颗粒粗大、流动性差、焊缝成形困难问题, 通过药皮设计形成复层梯度型熔渣可有效解决焊缝成形. 焊缝组织中, 抑制奥氏体柱状晶粗化、减小超低温条件下的塑性损伤是保证低温韧性的重要条件. 采用 ENiCrMo-6 型焊条焊接 9Ni 钢, 其焊态热影响区过热对板条马氏体和逆变奥氏体相的影响并不显著. 通常情况下, 焊接加热不致严重降低过热区中板条马氏体间残留逆变奥氏体相, 从而对过热薄弱区低温韧性具有组织保证作用.

创新点: (1) 创新性提出采用复层梯度型熔渣辅助高镍合金焊缝成形.

(2) 揭示出 ENiCrMo-6 镍基合金焊条异质焊接 9Ni 低温钢接头力学性能匹配的组织结构.

关键词: 镍合金焊条; 低温焊接; 熔滴过渡; 低温断裂; 力学性能

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0 序言

液化天然气 (liquefied natural gas, LNG) 作为一种清洁能源目前正在被迅速开发利用. 由于 LNG 储存和运输需在 -196°C 低温液化处理后进行, 为此 LNG 装备制造已成为其应用的重要基础保障. 20 世纪 40 年代, 美国率先研制出适用于 LNG 装备制造的 9Ni 低温钢, 其显微组织为回火马氏体, 经回火热处理可形成 $5\% \sim 15\%$ 的逆转变奥氏体, 可以获得理想的低温韧性及高强度力学性能, 奠定了 LNG 装备制造的材料基础^[1-2]. LNG 装备制造多在露天空间施焊, 一般情况下无需进行焊后热处理. 焊接工作者先后尝试了 9Ni 型焊缝、奥氏体不锈钢型焊缝等多种填充金属, 但基于镍基型焊缝的超级耐腐蚀性能以及 9Ni 钢在焊态强度、低温韧性和热膨胀系数方面的匹配性能, 是较为理

想的设计思路^[3-8].

镍基合金焊接不同于其它金属, 主要表现为液态粘度大, 流动性差, 熔池冶金反应不充分, 熔池中杂质元素难以充分上浮去除, 这些均可导致焊缝中气孔、夹渣和成形不良等缺陷. 同时, 镍基合金凝固后沿奥氏体单相区冷却不发生固态相变, 其晶粒长大现象十分突出^[4-6]. 国内应用的镍基合金焊接材料主要源于具有世界领先水平的美国 SMC 公司和 Techalloy 公司推出的系列镍基焊接材料. 此外, 英国、瑞典、德国以及日本等焊接材料公司也先后研发了镍合金关键焊接材料^[3]. 随着国内冶金技术的发展, 采用真空熔炼 + 电渣重熔二步法冶炼技术, 已在很大程度上解决了杂质元素的控制难题, 目前已基本能提供满足要求的超低杂质含量用填充镍材, 从而推动了镍合金焊接技术的发展. 国内一些科研院所和高等院校相继对镍基合金焊接技术开展过研究^[9-15]. 但镍基合金焊接作为高端装备焊接技术研究的重点领域, 其焊接过程中的冶金行为具有独特性. 由冶金特性决定的熔敷金属在深冷条件下的力学性能尚属目前焊接工作者科技攻关的课

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题. 因此有必要针对镍基异质材料填充低温钢焊接技术作深入研究.

1 试验方法

以 ENiCrMo-6 型焊条为研究对象, 根据国际规范对熔敷金属成分要求, 采用真空熔炼 + 电渣重熔

二步法冶炼制备出表 1 所示 $\phi 3.2\text{ mm}$ 镍材填充焊芯. 设计高碱度药皮配方, 经调试确定表 2 所示镍基焊条药皮配方基本组成. 将药粉干混均匀后加入模数 M3.1、KNa(1:1) 水玻璃湿搅拌, 在实验室用 T25 型焊条压涂机将药皮均匀涂敷在焊芯上, 再经 $380\text{ }^{\circ}\text{C} \times 1\text{ h}$ 烘焙即试制成 ENiCrMo-6 型镍基合金焊条.

表 1 ENiCrMo-6 焊条焊芯化学成分 (质量分数, %)
Table 1 Chemical compositions of core wire for ENiCrMo-6 electrode

C	Mn	Si	Cr	Ni	Mo	Fe	W	Cu	Nb + Ta	S	P
≤ 0.015	2.5 ~ 4.5	≤ 0.5	14.0 ~ 18.0	≥ 60	6.0 ~ 9.5	1.0 ~ 4.0	1.2 ~ 2.0	≤ 0.50	1.0 ~ 2.0	≤ 0.010	≤ 0.015

表 2 ENiCrMo-6 焊条药皮配方组成 (质量分数, %)
Table 2 Coating compositions of ENiCrMo-6 electrode

大理石	碳酸钡	萤石	氟化钡	钛酸钾	长石	金红石	金属合金
25 ~ 45	5 ~ 10	15 ~ 30	4 ~ 10	2 ~ 6	4 ~ 10	3 ~ 7	4 ~ 12

分别用 Q235 和 9Ni 钢试板对研制焊条进行熔敷金属力学性能和低温钢对接焊力学性能试验. 加工 2 块 $350\text{ mm} \times 200\text{ mm} \times 20\text{ mm}$ 的 Q235 钢板, 并各开 30° V 形坡口、根部间隙 5 mm, 坡口及垫板用待焊焊条堆焊 3 ~ 4 mm 厚稀释层. 由 2 块 $400\text{ mm} \times 250\text{ mm} \times 17\text{ mm}$ 的 9Ni 钢组成试板, 并各开 30° V 形坡口、根部间隙 2 ~ 3 mm. 经约 15 道多层焊制得熔敷金属测试和 9Ni 钢对接焊试样. 焊接过程中采用国产 Acuteye 高速摄影仪观察熔滴过渡形态及熔池流动状况. 焊后沿焊缝纵向取熔敷金属拉伸、冲击试样 (缺口位于焊缝中心); 沿焊缝横向取 9Ni 钢对接接头拉伸试样、弯曲试样、冲击试样 (缺口位于熔合区)、金相试样和透射电子显微镜分析薄片. 冲击试样经液氮浸泡 20 min 待温度冷却至 $-196\text{ }^{\circ}\text{C}$ 后立即进行低温冲击试验.

采用 JSM-6360LV 型扫描电子显微镜 (scanning electron microscope, SEM) 对拉伸、冲击断口进行形貌观察, 并配合能谱仪作微区成分和 X 射线衍射仪 (X-ray diffraction, XRD) 作相分析. 采用 OLYMPUS-GX51 型分析仪和 JEM-2100 型透射电子显微镜 (transmission electron microscope, TEM) 进行组织和晶体结构分析.

2 试验结果与分析

2.1 镍合金焊条熔滴过渡特征

图 1 为利用高速摄影仪测得的熔滴过渡过程照片. 高速摄像分析显示, 熔滴在焊条端部形成后可停留较长的时间, 以致长大至足够粗大颗粒尺寸, 在重力和电弧推力作用下才脱离焊条末端. 形

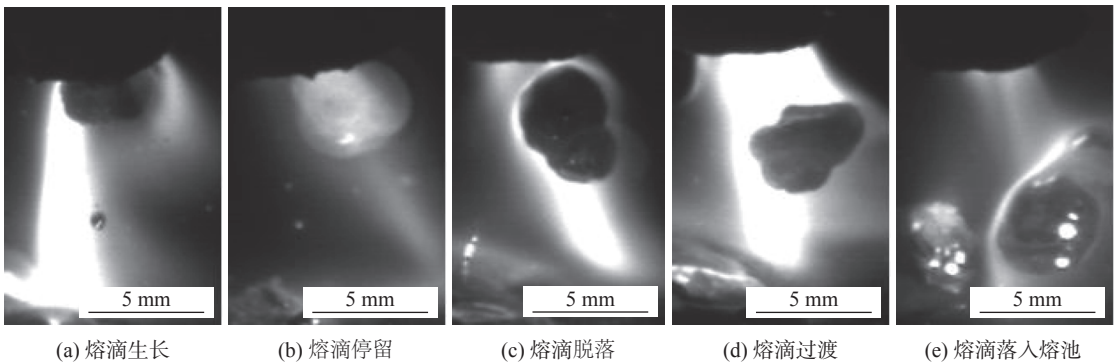


图 1 镍合金熔滴过渡高速摄影照片

Fig. 1 High speed photographs of droplet transfer for nickel alloy. (a) droplet growth; (b) droplet staying; (c) droplet separating; (d) droplet transferring; (e) droplet falling into weld pool

成的熔滴尺寸基本已达到焊条外径尺寸,即 5 mm 左右. 熔滴落入熔池后仍在一定时期内保持圆球形状,熔池中液体呈现较为明显的粘性流动特征. 上述观察现象与碳钢焊接熔滴过渡相比,液态镍合金由于粘性及表面张力大,形成熔滴时依靠表面张力作用在焊条端部可以停留更长的时间,易于形成粗大的熔滴. 考虑熔滴长大期间由于直接暴露于电弧空间,此时冶金反应最为激烈,氧化现象也因此十分严重. 这对 Ni, Cr, Mo 等稀贵金属的有效过渡极为不利. 为此镍合金焊接必须设计高碱度熔渣,以弱化气相及熔渣的氧化作用,否则难以保证有益合金元素的过渡从而影响力学性能. 同时,熔池中液态镍合金的高粘性对焊缝成形也带来极大的不利. 由于粘性大,液态金属流动性差,在很大程度上影响到熔池凝固的自然成形,也易于在焊缝边缘形成咬边缺陷. 常规通过调整熔渣成分,降低熔渣的表面张力,细化熔滴,改善润湿铺展性以获得焊缝成形,对碳钢焊接是有效的^[16-17],但对镍合金焊接而言,该技术具有一定的局限性.

2.2 复层梯度型熔渣铺展性能

为解决高碱度熔渣辅助焊缝成形,试验探索出采用渣相+铁合金复层型熔渣,可形成熔渣-液态铁合金-镍金属多层液态金属. 利用渣相表面张力显著低于铁液,且两相又互不相溶但却具有良好的润湿铺展作用,可辅助铁液表面成形. 而液态铁合金较液态镍表面张力小,因而又可形成分层并相互兼容. 当熔池中形成渣相-液态铁合金复合型熔渣与镍合金作用时,液态渣相可以较好地辅助液态铁合金成形,而液态铁合金又进一步铺展镍合金促使形成圆滑的焊缝表面. 该试验利用此原理成功地解决了镍合金焊缝成形差、脱渣困难技术难题. 利用高速摄像可以动态观察到复合型熔渣明显改善对高镍液态金属的润湿铺展性能,如图 2 所示,其凝固后覆盖焊缝及辅助焊缝成形照片如图 3 所示. 形成的凝固熔渣明显更为厚实坚硬,宏观上可观察到疏松渣相层沉积于硬壳表面. 进一步对固态熔渣断面 SEM 形貌观察发现存在明显的分层现象(图 4),线扫描分析表明,断面成分明显变化为富集铁合金的金属,而其余渣相的 XRD 分析结果主要为含 Ba, Ca, Al, Si 的氧化物渣相成分(图 5). 上述试验即可证实形成复合熔渣的客观事实.

2.3 熔敷金属的力学性能和微观组织

配合高碱度熔渣焊接获得的熔敷金属及异质

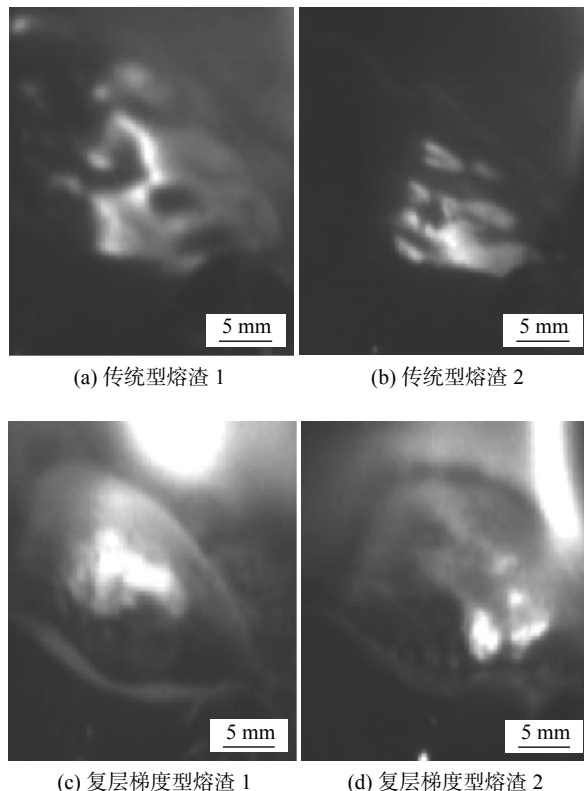


图 2 熔渣润湿铺展镍合金熔池高速摄像照片

Fig. 2 High speed photographs of molten slag wetting and spreading on nickel alloyed metal pool. (a) traditional molten slag 1; (b) traditional molten slag 2; (c) double gradient molten slag 1; (d) double gradient molten slag 2

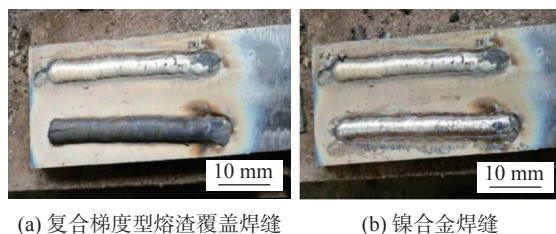


图 3 复层梯度型熔渣辅助镍合金焊缝形貌

Fig. 3 Double gradient molten slag assisted nickel base alloyed weld morphology. (a) double gradient molten slag covering on weld; (b) nickel base alloyed weld

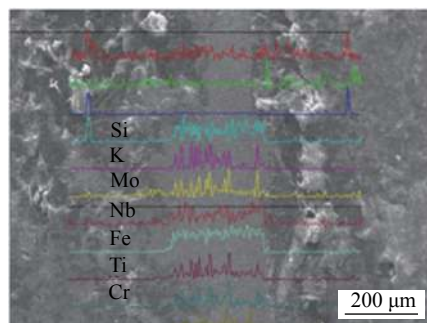


图 4 渣壳断面 SEM 照片

Fig. 4 SEM image of slag cross section

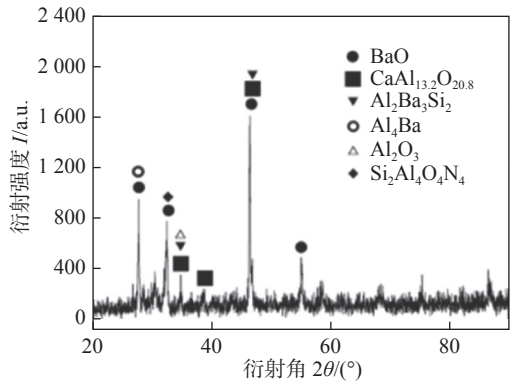


图 5 造渣相 XRD 图谱
Fig. 5 XRD patterns of slag phases

焊接 9Ni 钢对接接头力学测试结果分别如表 3 和表 4 所示. 结果表明, 熔敷金属及 9Ni 钢对接接头强度和塑性满足 NB/T 47014—2011《承压设备焊接工艺评定》要求, 且与 9Ni 钢有较好的力学性能匹配性, 尤其是可获得-196℃ 十分优异的超低温冲击韧性.

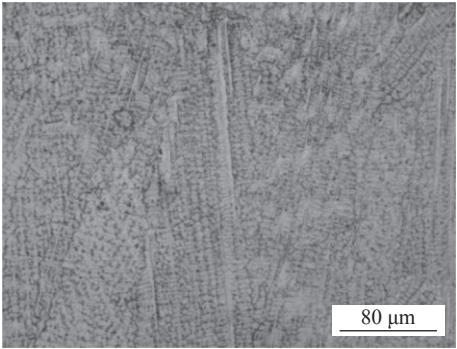
表 3 熔敷金属力学性能试验结果
Table 3 Mechanical properties results of deposited metal

项目	屈服强度 $R_{p0.2}$ /MPa	抗拉强度 R_m /MPa	断后伸长率 $A(\%)$	冲击吸收能量 (-196℃) A_{KV2} /J
要求值	≥410	≥620	≥27	≥47
实测值	458	702	45.5	123

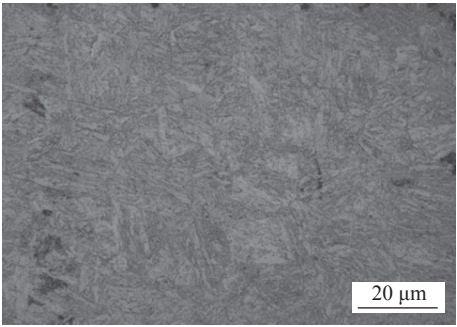
表 4 9Ni 钢接头的力学性能试验结果
Table 4 Mechanical properties results of 9Ni steel joint

项目	抗拉强度 R_m /MPa	面弯/背弯 ($\alpha = 180^\circ$)	熔合区冲击吸收能量 (-196℃) A_{KV2} /J	射线 探伤
要求值	≥620	无裂纹	≥47	I级
实测值	728	试样无开口缺陷	127	I级

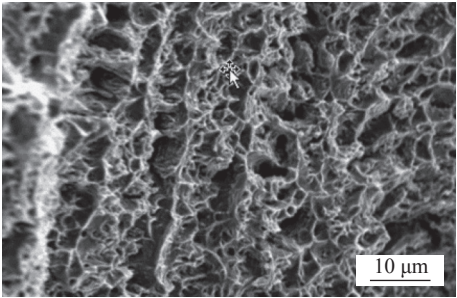
图 6 为试样的微观组织和断口形貌. 熔敷金属为典型的奥氏体凝固组织, 具有明显的柱状晶方向性, 如图 6a 和图 6b 所示, 而 9Ni 钢的热影响区呈现粗大的板条状马氏体. 为保证熔敷金属力学性能, 粗大柱状晶严重影响-196℃ 超低温韧性和断后伸长率. 当柱状晶宽度达到约 10 μm 以上时, 低温冲击值开始急剧下降, 呈现难以满足-196℃ 超低温技术要求特征. 这主要是由于镍合金热导率较低, 焊接电弧热量集聚焊接区域, 进而导致焊缝柱状晶生长过大、热影响区晶粒长大过快. 因此镍合金电弧焊有必要采用小热输入和冷却散热措施. 根据熔敷金属拉伸、冲击试样断口与低温冲击韧性值间的统计关系可验证上述结论. 从 9Ni 钢接头过热



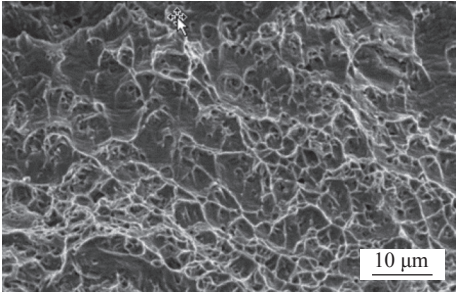
(a) 镍合金熔敷金属



(b) 接头热影响区



(c) 熔敷金属拉伸断口



(d) 熔敷金属冲击断口

图 6 试样的微观组织和断口形貌

Fig. 6 Microstructure and fracture morphology of specimens. (a) deposited metal of nickel alloy; (b) heat affected zone of joint; (c) tensile fracture of deposited metal; (d) impact fracture of deposited metal

区组织来看, 尽管存在较严重的组织粗化现象, 但能仍保持有较好的低温韧性. 这归结于钢中逆转变奥氏体组织对焊接过热脆化的抑制效应.

从图 6c 和图 6d 的断口特征可以看出, 无论是拉伸断口还是冲击断口, 均呈现韧窝型断裂. 拉伸

断口上仍可观察到焊缝柱状组织带状分布特征,这说明在静拉伸条件下裂纹首先沿柱状晶长度方向扩展.而 $-196\text{ }^{\circ}\text{C}$ 超低温冲击断口则表现为较浅的韧窝形貌,呈现舌状并具有一定准解理断口特征.试验表明镍合金奥氏体组织尽管具有很好地常温塑性,但超低温条件对镍合金塑性仍存在有一定的影响.因此镍合金超低温断裂与常温断裂相比,尽管均可表现出延性断裂,但超低温工况下的塑性损伤对断裂过程的裂纹扩展仍存在相当的影响.随着柱状晶增大对塑性的影响,则完全可以恶化为脆性断裂导致低温韧性失效.

2.4 低温韧性晶体学特征

为了从晶体学角度揭示组织在超低温条件下的初化机制,对镍合金熔敷金属和 9Ni 钢对接焊热影响粗晶区取样得到的薄膜试样进行透射电子显微镜组织结构观察.图 7 和图 8 为采用 ENiCrMo-6 焊条焊接 9Ni 钢接头中焊缝、熔合区和过热区薄膜透射形貌及晶体结构分析衍射花样.从 TEM 观察到 9Ni 钢接头中热影响区粗晶区精细结构为板条状形貌,内部分布有高密度的位错,衍射斑点分析主要为体心立方晶体,板条间发现尚存在有少量面心立方晶体结构.而焊缝衍射斑点为具有面心立方晶体学特征,几乎未发现有体心立方的铁素体晶

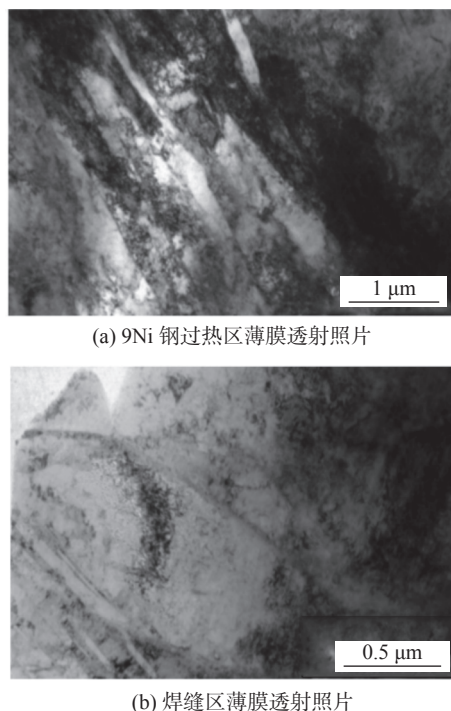


图 7 焊接接头 TEM 照片

Fig. 7 TEM photographs of welded joint. (a) film transmission photograph of over heating zone; (b) film transmission photograph of weld metal

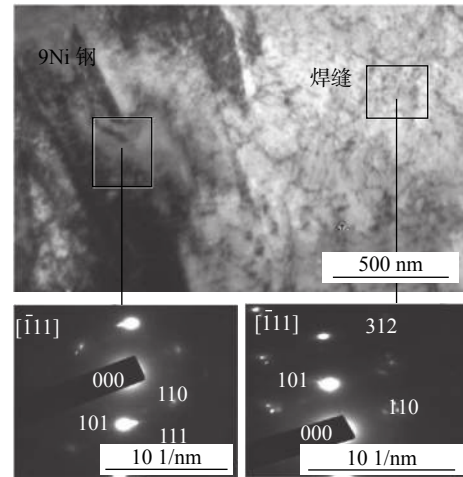


图 8 熔合区薄膜透射及相结构衍射花样照片

Fig. 8 Film transmission photograph of fusion zone with diffraction pattern for phase structure

体结构.焊缝和热影响区板条间具有相同的晶体学特征.因此可以判定热影响区主要为板条马氏体和少量的逆变奥氏体.尽管板条马氏体受焊接过热严重长大,但由于其中逆变奥氏体的存在,仍可以较好地保证其低温韧性.这表明焊接高温残留的逆变奥氏体的存在是保证过热区韧性的关键.

3 结论

(1) 镍合金具有粘性高、表面张力大本征特性,焊接时可显著增大熔滴长大周期,呈现粗颗粒过渡,并使液态熔池流动成形性差.采用复层梯度型熔渣是解决辅助镍合金焊缝成形的一有效技术措施.

(2) 研制的高碱度镍合金焊条熔敷金属及异质填充焊接 9Ni 钢具有很好的力学性能匹配,尤其是具备优异的 $-196\text{ }^{\circ}\text{C}$ 低温性能.焊缝组织中抑制奥氏体柱状晶粗化、减小超低温条件下的塑性损伤是保证低温韧性的关键.

(3) 用镍合金异质填充焊接 9Ni 钢,采用小焊接热输入施焊,尽管其热影响区中逆变奥氏体存在过热长大现象,但对过热区韧性降低并不显著.一般条件下,可以确保过热区中板条马氏体间残留一定比例的逆变奥氏体相,从而保证过热区的低温韧性.

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Key words: parabolic model; welding bead planning and layout; equal area method; equal height method; MATLAB simulation; multi-layer and multi-pass welding

Droplet transfer characteristics and mechanical properties of deposited metal as welded matching with cryogenic temperature steel for nickel based consumables

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Abstract: The ENiCrMo-6 electrode for cryogenic temperature application was developed which was made of nickel alloy core wires obtained by duplex refining of vacuum melting and electroslag remelting, and specially designed coatings with high basicity allowing the formation of gradient composite slag. Mechanical property tests were carried out on the deposited metal and the welding joint of 9Ni steel welded by ENiCrMo-6 electrode. With the aid of Acuteye high speed photograph technology, the characteristics of the droplet transfer and the molten metal flow have been studied, and the microstructure of the composite slag layer has been observed by JSM-6360LV scanning electron microscope. The weld and heat affected zone are observed and analyzed to investigate the low-temperature fracture behavior from different perspectives. The results show that large shaped molten droplets of nickel alloy and the poor fluidity result in difficulties in weld formation, while special designed coatings is an effective solution, which allows the formation of a gradient composite slag layer to accelerate the forming of weld. To ensure joint ductility in cryogenic conditions, it is critical to avoid the coarsening of austenite columnar grains and reduce the ductility loss in weld metal as welded. The experiment results has proved that when welding 9Ni steel with ENiCrMo-6 electrode, the effect of overheat on lath martensite or reversed austenite in the as-welded heat affect zone is insignificant. Generally, overheat will not largely reduce the number of reversed austenite or retained austenite within the lath martensites, which guaranteed the ductility of joint under cryogenic condition.

Highlights: (1) Gradient composite slag layer was innovatively put forward to assist the formation of high nickel alloy weld.

(2) The mechanical property of welding joint obtained by low temperature welding of 9Ni steel with ENiCrMo-6 electrode

highly matches with that of the base metal.

Key words: nickel-alloy electrode; cryogenic welding; droplet transfer; cryogenic fracture; mechanical properties

Microstructure and properties of 7075-T651 aluminum alloy friction plug welding joint

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Abstract: Friction plug welding (FPW) for 8 mm thickness 7075-T651 aluminum alloy sheets were carried out using 2219-T87 aluminum alloy as plug material, and the formation and microstructure characteristics of the FPW joints obtained by using different process were observed, and the properties were tested. The results show that defect-free joints can be obtained with optimized parameters, and equiaxed grains were formed at the friction interface between the plug and base material. According to the structure and morphology, the FPW joint can be divided into 6 zones: recrystallization zone, thermo-mechanical affected zone, plug thermo-mechanical affected zone, heat affected zone, plug heat affected zone and base metal. The microstructure of the thermo-mechanical affected zone grew and deformed, with the precipitation phase dissolved and coarsened. Local softening also occurred. In particular, the hardness value of the plug heat affected zone reached the lowest at about 90 – 95 HV. During the tensile test, fracture of the defect-free FPW joint happened at the plug center, with ductile features in terms of fracture mode. The maximum tensile strength and elongation of the FPW joints reached 343 MPa and 7.0%, reaching 63.8% and 77.8% of that of the base metal, respectively.

Highlights: (1) The main types and causes of welding defects were investigated through the study of FPW process and microstructure of 7075-T651 aluminum alloy.

(2) The relationship between the degradation of mechanical properties of FPW joints in different regions and the microstructure morphology and precipitation phase distribution was revealed.

Key words: friction plug welding; 7075-T651 aluminum alloy; microstructure; precipitation phase; mechanical property