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银钎剂水溶法改性制备工艺的优化

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摘 要：针对银钎剂生产成本较高、性能不够稳定以至难以大批量生产的现状，通过在银钎剂的水溶法改性工艺的基础上，采用正交试验法对其工艺及配方进行了优化设计。正交试验表明，烘干时间对钎剂性能的影响最为显著，其次是成分配比，再次是烘干温度。水溶法改性最佳方案是，钎剂配比为 42% KF + 23% KBF_4 + 35% B_2O_3，烘干时间为 45 min，烘干温度为 350 ℃。实施了优选的工艺其操作方便、生产成本低、产品质量稳定。

关键词：银钎剂；正交试验；吸湿性；熔点；铺展性
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0 序 言

银钎剂由氟化钾、硼化物和复杂的氟化物组成[1]。国内应用率达 80%的 QJ102成分是由 35% B_2O_3、42% KF和 23% KBF_4组成。含大量 KF的银钎剂活性强，寿命长，适用于各种钎焊方法。由于含 KF的粉末状银钎剂开瓶后容易潮解结块不能正常使用，从而造成极大的浪费。为了提高产品性能，降低钎剂的吸潮性和改善调膏性，熔凝法、烧结法和水溶法等改性途径不失为银钎剂改性处理的有效手段。但在银钎剂生产实践过程中，水溶法具备反应时间短且反应温度低的特性，有着生产效率高、生产能耗低、操作方法方便等诸多优点[2]。通过在水溶法基础上采用正交试验法，对银钎剂制备的工艺参数进行优选，并在优选的工艺参数下重复试验，从而获得了质量稳定、粒度均匀、吸湿性小的产品。

1 钎剂的配制

根据 QJ102配方和水溶法改性处理工艺的要求，钎剂的吸湿性、熔点和铺展性作为考察指标，成分配比、烘干时间、烘干温度作为三个因素 A、B、C，并各选三个水平，如表 1所示。正交试验[3]方案及其结果如表 2所示。

表 1 试验因素水平表

Table 1 Experimental factors and levels

因素/水平	A 成分配比 (质量分数，%)	B 烘干时间 t/min	C 烘干温度 T/℃
1	40% KF + 25% KBF_4 + 35% B_2O_3	35	350
2	42% KF + 23% KBF_4 + 35% B_2O_3	45	400
3	44% KF + 21% KBF_4 + 35% B_2O_3	55	450

表 2中综合评分 Y数据的计算过程如下。

试验指标综合加权评分

$$Y_i = b_{i1}Y_{i1} + b_{i2}Y_{i2} + \cdots b_{ij}Y_{ij}, \tag{1}$$

式中：b_{ij}表示系数；Y_{ij}表示试验指标；i表示第 i号试验；j表示第 j个试验指标。

其中 b_{ij}确定方法是，设权重 100分为满分，吸潮性占 40分，相对熔点占 20分，铺展面积占 40分。

$$b_{ij} = \frac{分值}{K}, \tag{2}$$

式中：K为各试验指标的变化范围，即最大值与最小值之差为

K_1(吸潮增重) $= 0.9599 - 0.0794 = 0.8805$;

K_2(相对熔点) $= 570 - 528 = 42$;

K_3(铺展面积) $= 130 - 92 = 38$.

可得 $b_{i1} = 40/0.8805 = 45.4$,

$b_{i2} = 20/42 = 0.48$,

$b_{i3} = 40/38 = 1.05$.

计算综合加权评分值，回潮率以 0为基准，取正；相对熔点以 500 ℃为基准，取正；吸湿铺展面积以 100 mm^2 为基准，取负。

则 $Y_1 = 45.4 \times 0.1058 + 0.48 \times 28 - 1.05 \times 0 = 18.24$;

$Y_2 = 45.4 \times 0.473 + 0.48 \times 61 - 1.05 \times 30 = 19.25$;

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表 2 正交试验方案与结果

Table 2 Projects and results of orthogonal test

因素 / 试验号	成分配比 A 1	烘干时间 t/min 2	烘干温度 T/℃ 3	回潮率 5天 (%)	相对熔点 T/℃	铺展面积 A/mm²	综合评分 Y
1(S_{111})	1	1	1	10.58	528	100	18.24
2(S_{122})	1	2	2	47.30	561	130	19.25
3(S_{133})	1	3	3	95.99	560	95	77.63
4(S_{223})	2	2	3	9.92	528	110	7.43
5(S_{231})	2	3	1	8.30	560	130	1.07
6(S_{212})	2	1	2	7.94	570	110	26.71
7(S_{332})	3	3	2	11.09	560	92	42.24
8(S_{313})	3	1	3	9.88	534	125	−5.44
9(S_{321})	3	2	1	10.48	530	110	8.66
I	115.12	39.51	27.97	—	—	—	—
II	35.21	35.34	88.20	—	—	—	—
III	45.46	120.94	79.62	—	—	—	—
R	79.91	85.60	60.23	—	—	—	—
较优水平	2	2	1	—	—	—	—
因素主次		B→A→C					

$$Y_9 = 45.4\times0.1048+0.48\times30-1.05\times10=8.66.$$

2 试验方法

2.1 吸湿性试验

吸湿性是钎剂性能的一项重要指标。将表 2中所设计的试验方案所制备的银钎剂置于通风处，每天固定时间用分析天平称重，测定它们的增重情况(温度 25～28 ℃，湿度 60%～70%)。

2.2 测定熔点的试验

使用美国 PA公司的 SBT2960热重分析仪对研制合成的钎剂进行熔点测试，升温速率为 15 ℃/min。

2.3 铺展性能的测试试验

按照 GB 11364—89《钎料铺展性及填缝性试验方法》，将已制备好的银钎剂配合 BAg56CuZnSn银钎料，在 720 ℃下对 40 mm×40 mm×3 mm 的紫铜片进行铺展性试验。

3 试验结果与分析讨论

3.1 正交试验结果

由正交法的方差分析得出各因素的主次顺序为 B→A→C，即烘干时间对钎剂性能的影响最大，其次为钎剂的成分配比，再次为烘干温度，最优试样为 S_{221}，即钎剂含量为 42% KF + 23% KBF_4 + 35% B_2O_3；烘干时间为 45 min，烘干温度为 350 ℃。据此方案重复试验三次，效果均良好。

3.2 钎缝宏观分析

以优化后的银钎剂 S_{221}为助钎剂，钎料为银基钎料(标准号 GB 10046－88，型号 BAg45CuZnCd)，在室温下对紫铜条进行钎接，钎缝宏观图如图 1所示。采用此方案的钎剂具有优良的润湿性和流动性，钎缝的起焊端和收尾端均呈凹弧状，无气泡，钎料易进入钎缝里，钎料在紫铜基体表面发生铺张，无疙瘩，钎接后的残渣清除方便。

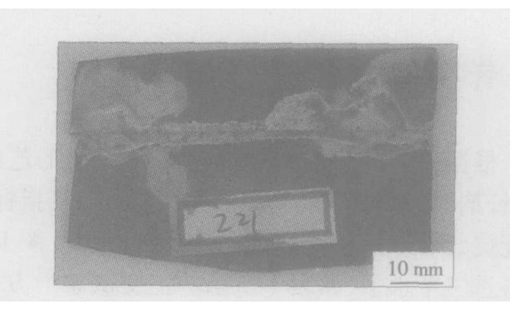

图 1 钎缝的宏观照片

Fig. 1 Result of brazing seam

3.3 钎缝的金相组织

钎缝与母材交接线为一条较分明的细线，这是

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中存在互反射和表面遮挡。

(3) 熔池表面的光滑模型由二阶光滑部分和边缘零阶连续部分组成, 用可控样条函数的离散形式表示。

(4) 计算出来的表面高度值反映了铝合金熔池的形状特点。

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钎料和母材的熔合线, 也就是钎缝。由图 2可清楚看到钎缝不仅界面致密, 而且均匀连续。

3.4 银钎剂的 X射线衍射结构分析

用 S_{221}方案制备的钎剂的 X射线衍射结构分析结果为 $KBF_4+K_2[(OH)F_4B_3O_3]+K_2B_3O_3F_4OH+KBF(OH)_3$, 显然经水溶法改性处理后, 钎剂的化学物质组成发生了变化。其中 KBF_4 微溶于水, 无吸湿性[4], $K_2[(OH)F_4B_3O_3]$ 也不吸潮, 而 $KBF(OH)_3$ 相对前两者吸潮较大, 但也远小于 KF。所以银钎剂改性处理后吸湿性大大减小。

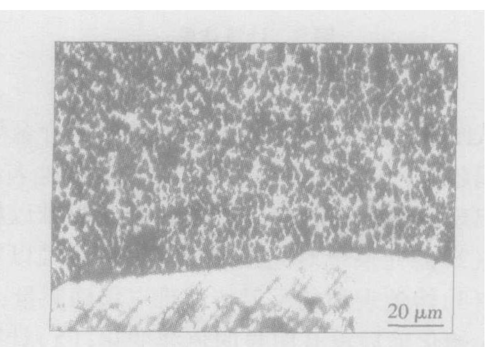

图 2 钎缝取样的金相照片

Fig. 2 Micrograph of brazing seam

4 结 论

通过正交试验法对银钎剂的水溶法改性处理的参数进行优化设计, 发现烘干时间对钎剂综合性能的影响最为显著, 其次是钎剂的成分配比, 再次是烘干温度。水溶法改性处理的最佳方案是, 钎剂含量为 42% KF + 23% KBF_4 + 35% B_2O_3; 烘干时间为 45 min; 烘干温度为 350 ℃。采用这种方案制得的钎剂质量稳定、粒度均匀、吸湿性小, 钎剂在钎焊过程中具有优良的润湿性、流动性、铺展性。生产工艺操作方便、生产能耗低、环境污染少, 有利于批量生产, 推广应用有着显著的经济效益和社会效益。

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welded by using scanning heating technology to prepare the powder metallurgical transition layer.

Key words: electron beam (EB); scanning control system; scanning track; energy control

Diffusion bonding of TC4/Ni/QAl10-3-1.5 GUO Wei[1], ZHAO Xi-hua[1], SONG Min-xia[1], FENG Ji-cai[2], YANG Biao[1] (1. School of Material, Jilin University, Changchun 130025, China; 2. State Key Laboratory of Advanced Welding Production Technology, Harbin Institute of Technology, Harbin 150001, China). p63 – 66

Abstract: TC4 and QAl10-3-1.5 were diffusion-bonded with Ni interlayer. The diffusion-bonded joints were evaluated by scanning electron microscopy (JEOL JSM 6700F) and its attaching energy dispersive spectroscopy (EDS). Intermetallic compounds at the interface were detected vie X-ray diffraction (XRD) and microhardness tests and tensile testings were done to evaluate the properties of bond joints. The results indicated that TC4 and QAl10-3-1.5 with Ni interlayer were bonded firmly under the condition of 870 ℃ temperature, 10MPa bond stress and 60min holding time, and the bond strength was up to 325MPa, furthermore, various reaction bands appeared in the diffusion zone, and NiTi phase (NiTi+ Ni_3Ti) phase and Ni(Cu) solid solution were produced at the interface zone.

Key words: diffusion bonding; titanium alloy; copper alloy; Ni interlayer; microstructure

Soldering of LD31 aluminum alloy with electro brush plated Sn-Pb alloys ZHAO Zhen-qing[1], WANG Chun-qing[1], DU Miao[2], HUANG Yi[1] (1. Harbin Institute of Technology, Harbin 150001, China; 2. Harbin Welding Institute, Harbin 150080, China). p67 – 70, 74

Abstract: The bonding of LD31 aluminum alloy with electron brush plated Sn-Pb solder after the deposition of Ni and Cu transient layers was investigated in this paper. The influence of electric deposition parameters of SnPb alloy on the coating quality and subsequent solderability was studied. The constitution of coatings, the morphology of Sn-Pb coating, the bonding mechanism in the soldering and the elements distribution in the soldered joint were studied by scanning electron microscope, energy dispersive X-ray analysis and metallographic analysis. The results of soldering experiment showed that after the deposition of Ni and Cu coatings, the bonding of LD31 aluminum alloy could be transformed to the bonding of copper, and the bonding of coatings could bear the heating condition in the soldered. The shear strength of the soldered joint could reach as high as 20MPa.

Key words: LD31 aluminum alloy; electron brush plating; coating; soldering

Particularity analysis of Francis turbine runner's simulation during welding JI Shu-de, FANG Hong-yuan, LIU Xue-song, MENG Qing-guo (Harbin Institute of Technology, Harbin 150001, China). p71 – 74

Abstract: Aiming at the particularity of Francis turbine runner's simulation in the process of welding, the method was adopted which solved the loading of welding thermal source by means of node-to-node connection and the method was brought forward which solved the problem of weld's wire feed by dividing the weld into many parts. These two methods were proved reasonably by the runner's temperature field. Moreover, the problem of heat conduction between independent entities and the problem of heat elimination between blade and air were successfully solved by using contact bodies. It had important guiding significance to the numerical simulation of complicated weldments.

Key words: runner; welding; numerical simulation; temperature field

Optimization of water dissolution modification process using silver brazing fluxes CHEN Hai-yan, LI Wei-min, LI Feng, SHU Chang (Faculty of Material and Energy, Guangdong University of Technology, Guangzhou 510643, China). p75 – 76, 80

Abstract: The optimization of the water dissolution process using silver brazing fluxes was conducted by orthogonal test. The test result showed that effect of bake time on flux property is the best notable, next proportioning of compositions affected flux property, then bake temperature affected flux property. The optimum process parameters are component of 42% KF+23% KBF_4 +35% B_2O_3, bake time of 45min and bake temperature of 350 ℃. By using the silver brazing fluxes, the best results of the jointing can be carried out.

Key words: silver brazing flux; orthogonal test; absorptivity; melting point; spreading property

Modeling of welding pool surface reflectance of aluminum alloy pulse GTAW LI Lai-ping, CHEN Shan-ben, LIN Tao (School of Materials Science and Engineering, Shanghai Jiaotong University, Shanghai 200030, China). p77 – 80

Abstract: The key to the surface height calculation of pulse GTAW welding pool based on shape from shading (SFS) is to build up a surface model of welding pool. Based on the imaging characteristics of aluminum alloy pulse GTAW welding pool, the surface reflectance model is built after analyzing arc intensity, filter system, welding pool shape and reflectance characteristics. With smooth constraint condition of the welding pool surface and variable factor successive over relaxation (SOR) method, the height of welding pool surface is calculated and the error is analyzed.

Key words: aluminum alloy pulse GTAW; surface reflectance model of welding pool; shape from