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CORRELATION BETWEEN MICROSTRUCTURE AND MECHANICAL PROPERTIES OF DIFFUSION BRAZED MAR-M 247

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ABSTRACT

Diffusion brazing is a joining process utilized in the manufacture and repair of turbine blades and vanes. MAR-M247 is an investment cast Ni-based superalloy used for turbine blading and has good strength properties at high temperatures. The objectives of this work was to develop a diffusion brazing procedure to achieve high strength joints. A commercially available diffusion brazing filler metal of composition Ni-15Cr-3,5B of 100 μ m thickness was used. With the desire to eliminate brittle centre-line phases, the effects of the processing variables (only temperature and time) on the joint microstructure was studied. Once the metallurgy of the joint was understood, mechanical property assessments were undertaken i.e. tensile and creep rupture tests, and the latter being the severest test to evaluate joint strength. The results demonstrated that the diffusion brazed joints had nearly equivalent mechanical strength to that of the parent metal. This showed that the resultant diffusion brazing parameters enabled effective and reliable joining of MAR-M247.

INTRODUCTION

The CSIR, a research organization, was approached with a requirement to change/modify nozzle guide vane assemblies from an all cobalt based alloy to Ni-base superalloy assemblies to reduce fuel consumption and increase thermal efficiency by 4 to 5 %. In the manufacture of such a Ni-base superalloy nozzle guide vane assembly, MAR-M247 was chosen as the replacement vane metal because of its good strength at high temperature. These vane aerofoils had to be joined to the inner and outer rings. Due to the high Al and Ti content and thus its high volume fraction of γ' , a $\overline{Ni_3}$ (Al, Ti) phase, MAR-M247 is extremely difficult to weld due to problems such as hot cracking and HAZ micro-fissuring (Owczarski, 1977 and Duvall and Owczarski, 1967).

Finite element analysis also revealed that the maximum stress occurred at the joint interface and hence the joining technique used had to achieve good, high strength joints.

The improved application and success of the diffusion brazing process world-wide (Duvall et al, 1974 and Hoppin and Barry, 1970) with Ni-base filler metals resulted in a research programme being initiated to join MAR-M247 vane aerofoils to the inner and outer rings. Diffusion brazing is a process involving coalescence of metal using an interlayer filler metal which melts and joins the faying surfaces together. The brazed assembly is held at the brazing temperature for sufficient time for diffusion to produce a nearly uniform alloy composition across the joint. In actual fact the filler metal is diffused into the parent metal to an extent that a distinct layer of braze filler does not exist in the joint after the diffusion brazing process is completed.

A second important feature was to develop a possible repair scheme in which thermal fatigue cracks on the aerofoils could be repaired.

EXPERIMENTAL PROCEDURE

Investment cast MAR-M247 samples were made available for this work. The composition of this alloy is Ni-lOCo-1OW-8,25Cr-5,5A1- lTi-3Ta-0,7Mo-0,2Si-0,5Fe- 1 , 5Hf-0, 05Zr-0,015B-0,15C. The composition of the Nicrobraz 150 brazing filler metal is Ni-15Cr-3,5B and is commercially available from Wall Colmonoy. The filler metal was in transfer tape form and was $100 \mu m$ thick. It should be noted that the filler metal was not ordered to any specification.

Test sample faying surfaces were polished up to a 1 200 emery grit paper and then cleaned with toluene and ethanol. The joint gap was maintained at 100 μ m by placing two, 100 μ m molybdenum foil sections between the faying surfaces as seen in Figure 1.

A binder was used to hold the filler metal in place. All diffusion brazing experiments were conducted in a Lindberg tube furnace in a vacuum of 5×10^{-5} mBar. Brazing was carried out at temperatures from 1 125 °C to 1200 °C for certain lengths of time as shown in Table 1.

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Table 1: Temperatures and times used for brazing experiments

TEMP (°C)	TIME (mins)								
1125° C	20	60	120	X	240	x	x	X	x
1150° C	20	60	120	X	X	360	x	x	x
1200°C	20	60	120	180	x	360	3540	10080	14400

No formal design of experiments was used to determine the braze temperatures and times shown in Table 1. The lowest temperature (1125 °C) is the suggested brazing temperature recommended by the manufacturer. An alternate aim of recommended by the manufacturer. performing the experiments shown in Table 1, was to analyze the effects of an increase in time at a constant temperature and increase of temperature at a constant time.

For mechanical testing, after diffusion brazing, all samples were solution heat treated at 1 250 °C for 4 hours at 50 MPa and aged at 870 °C for 20 hours. The same heat treatment was given to the parent metal/baseline samples in all cases. The heat treatment at 1 250 °C for 4 hours at 50 MPa was in actual fact a combination of a solution heat treatment and a hot isostatic pressing heat treatment. The use of the 50 MPa pressure was primarily to close up porosity in the parent and braze metal regions.

After brazing and subsequent heat treatment, all braze joints were inspected prior to cut-up using a real time microfocus X-ray machine. Thereafter samples were sectioned and prepared using standard metallographic preparation techniques. The etchant used was Marbles reagent.

METALLOGRAPHIC RESULTS AND DISCUSSION

Figure 2 shows the microstructure of the joint after brazing at 1 125 °C for 20 minutes. A continuous, dense, hard and brittle centre-line phase existed which is undesirable as it provides a continuous path for crack propagation. Figure 3 shows the microstructure of the joint after brazing at 1 125 °C for 60 minutes. The continuous centre-line phase which existed previously had partially disintegrated forming a semi-continuous boride phase consisting of large and small particles.

Figure 1: Schematic representation of braze sample set up.
Figure 2: $T = 1.125 °C$; t = 20 minutes
Mog 90x Mag 80x

Figure 3: $T = 1 125 °C$; $t = 60$ minutes Mag 80x

Figure 4 shows the microstructure of the joint after brazing at 1 125 °C for 120 minutes. The semi-continuous centre-line phase still existed. The width of the centre-line phase has partially diminished and the individual boride particles have also decreased in size. Figure 5 shows the microstructure of the joint after brazing at 1 125 °C for 240 minutes. Clearly the centre-line phase has disintegrated a great deal and only isolated particles are present.

Figure 6 shows the microstructure of the joint after brazing at 1 150 °C for 20 minutes. As was the case with the joint brazed at 1 125 °C for 20 minutes (Figure 2), a large continuous centre-line phase existed. Figure 7 shows the microstructure of the joint after brazing at $1\,150\,^{\circ}\text{C}$ for 60 minutes. The width of the centre-line phase has decreased marginally but it is still continuous. Boride needle-like phases can also be seen at the joint interface as a result of B diffusing away from the joint into the parent metal.

Figure 8 shows the microstructure of the joint after brazing at 1 150 \degree C for 120 minutes. The formation of grains in the joint area has occurred. Figure 9 shows the microstructure of the joint after brazing at 1 150 °C for 360 minutes. In comparison with previous figures, clearly a tremendous change in the microstructure of the joint has occurred. The Ni-solid solution area (i.e. the area adjacent to the centre-line phase) and the joint interface has totally disintegrated as a result of both diffusion of elements from the parent metal into the joint and vice versa.

The eutectic and carbide structures evident in this figure resemble in morphology those present in the parent metal microstructure, hence there is difficulty in detecting the joint area.

Figure 4: $T = 1 125 °C$; $t = 120$ minutes Mag 150x Mag 80x

Figure 5: $T = 1 125^{\circ}C$; t = 240 minutes Mag 100x

Figure 6: $T = 1 150 °C$; $t = 20$ minutes Mag 50x

Figure 7: $T = 1 150 °C$; $t = 60$ minutes Mag 150x

Figure 8: $T = 1 150 °C$; $t = 120$ minutes

Figure 9: $T = 1$ 150 °C; t = 360 minutes Mag 50x

Figure 10 shows the microstructure of the joint brazed at 1 200 °C for 20 minutes. As in the case in Figures 2 and 6, when brazing for 20 minutes, a large continuous centre-line boride phase existed. Figure 11 shows the microstructure of the joint brazed at 1 200 °C for 60 minutes. A very narrow centre-line phase existed. This phase does not run planar but branches like a river with its tributaries towards the joint interface. Boride needle-like phases were also present at the joint interface as was the case when brazing for 60 minutes at $1\,150\,^{\circ}$ C (Figure 7).

Figure 12 shows the microstructure of the joint brazed at 1 200 °C for 120 minutes. The narrow centre-line phase still existed, however the Ni-solid solution area and the joint interface have disintegrated due to diffusion of elements from the parent metal into the joint. Figure 13 shows the microstructure of the joint brazed at 1 200 °C for 180 minutes. The centre-line phase has now become discontinuous. Eutectic phases have formed along the boride centre-line phase as well.

Figure 14 shows the microstructure of the joint brazed at 1 200 °C for 360 minutes. The centre-line phase has totally disintegrated. The only means of detecting the joint area was to look at higher magnifications for eutectic and carbide phases which resembled eutectic and carbide phases found along the grain boundaries of the parent metal microstructure.

Figure 10: $T = 1200 °C$; $t = 20$ minutes Mag 50x

Figure 11: $T = 1200$ °C; $t = 60$ minutes Mag 150x

Figure 12: $T = 1200 °C$; $t = 120$ minutes Mag 50x

Figure 13: $T = 1200$ °C; $t = 180$ minutes Mag 50x

Figure 14: $T = 1200$ °C; $t = 360$ minutes Mag 500x

As evident in Figure 14, the joint area was becoming indistinguishable from the parent metal. Thus as time is increased it was realised that detecting the joint and analysing the microstructure may become a difficult task. Thus electrolytic etching with H_3PO_4 was used for the following micrographs.

Figure 15 shows the microstructure of the joint brazed at 1 200 °C for 59 hours (3 540 minutes). Clearly no centre-line boride phase, carbide or eutectic phases were present in the joint. However boride needle-like phases were present in the parent metal and γ ' precipitates (although not clearly visible in Figure 15) were present in the joint area. The mechanism for this phenomenon is that B has diffused away from the joint into the parent metal and formed boride needle-like phases. Also Al and Ti diffused from the parent metal into the joint to form the fine γ' phase. The boride needle-like phases were actually W-Cr-Ni boride phases as confirmed by Auger analysis as seen in Figure 16(a). Carbides were also found in the joint and these were Ta-Hf-W rich as evident in the Auger analysis of Figure 16(b).

Figure 15: $T = 1200 °C$; $t = 59$ hours (3 540 minutes) Mag 200x

The centre-line phase of all the joints shown in Figures $2\rightarrow 14$ generally consisted of both Cr boride phases, as seen in the Auger analysis of Figure 16(c), and Ni-Cr-Co-Al-W boride phases as seen in Figure 16(d).

Figure 16(a): Auger analysis of needle-like phase

Figure 16(b): Auger analysis of carbide phase

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Figure 16(d): Auger analysis of centre-line phase

Figure 17 shows the microstructure of the joint after brazing at 1 200 °C for 168 hours (1 0080 minutes). The formation of equiaxed grains in the joint had occurred. No boride phases were found present in the joint and parent metal. As can also be seen in Figure 17, microporosity is evident in both the parent metal and joint, hence it was decided that a post-braze heat treatment involving 'hipping' would be carried out The use of a real time microfocus X-ray machine revealed microporosity in the joint as well as the parent metal. The voltage and current used to detect defects in the joint was 130 kV and 0,9 mA. Although not evident in the figures, the levels of porosity was higher in the parent metal than in the joint. However, both levels of porosity were acceptable using the South African standard which is that the reference area for area percentage is 1 mm x 1,4 mm. The maximum value for microporosity area percentage is 0,5 %. For gas pores (cavities) the maximum allowable size of a single pore, depending on the location, is 3 mm. The distance between 2 pores must be at least 12 mm in case of hollow blades and at least 8 mm in case of solid blades. Figure 18 shows the microstructure of the joint after brazing at 1 200 °C for 240 hours (14 400 minutes). The equiaxed grains in the joint were still present with carbide phases along these grain boundaries. This is an ideal situation as one has equiaxed grains in both the parent metal and joint area. The volume fraction of γ' in the joint was 59 % and 62 % in the parent metal. The volume fraction was measured by using a software program on an image analyzer facility.

Figure 16(c): Auger analysis of centre-line phase Figure 17: $T = 1200 °C$; $t = 168$ hours (1 0080 minutes) Mag 200x

Figure 18: $T = 1200 °C$, $t = 240$ hours (1 4400 minutes) Mag 200x

Thus summarising both an increase in time and temperature play a beneficial role in improving the homogeneity of the joint microstructure. The joint is homogenised when diffusion brazing for periods of days resulting in the formation of an equiaxed microstructure in the joint area similar to that present in the parent metal. The volume fraction of the γ' phase is nearly equivalent to that of the parent metal.

MECHANICAL PROPERTIES

As can be clearly seen in the previous paragraphs a large range of microstructures existed for a variety of brazing times and temperatures. The obvious question is what properties do these microstructures have. Hence tensile tests at room temperature and 760 °C as well as creep rupture tests at 980 °C were carried out. Tensile tests were conducted based on the tensile test butt configuration shown in Figure 19(a). Creep rupture tests were conducted based on the configuration shown in Figure 19(b). The two test configurations shown are a 100 % full penetration butt joint. All mechanical property results are an average of 3 samples, or 2 samples where otherwise stated.

Figure 19(a): Tensile strength test configuration for brazed joints

Figure 19(b): Creep rupture strength test configuration for brazed joints

Figure 20 shows the tensile stress variation at room temperature as a function of brazing parameters for butt joints with Nicrobraz 150 filler metal. After brazing at 1 125 °C/30 minutes, the joint failed at an U.T.S. of 443 MPa. It was deduced that the low U.T.S. value resulted from the easy crack propagation path provided by the continuous centre-line phase as seen in Figure 2. Increasing the brazing temperature and time to 1 150 °C/120 minutes, the joint failed at an U.T.S. of 720 MPa. The increase in strength of the joint is due to the formation of equiaxed grains in the joint (Figure 8) and the disintegration of the continuous centre-line phase. Increasing the temperature and time still further to 1 200 °C/360 minutes, the parent metal now failed at an U.T.S. 918 MPa. It can be recalled that the joint area resembles in morphology that of the parent metal (Figure 14). Hence the joint area is homogenised after brazing at 1 200 °C for 360 minutes. This briefly explains why parent metal tensile strength was achieved.

Figure 21 shows the tensile stress variation at room temperature as a function of diffusion brazing parameters for butt joints with Nicrobraz 150 filler metal. After diffusion brazing at 1 200 °C/30 hours the parent metal failed at an U.T.S. of 960 MPa. Increasing the diffusion brazing time still further to 1 200 °C/3 days, the parent metal failed at an U.T.S. of 966 MPa. Finally increasing the time to 1 200 °C/5 days, the parent metal failed at an U.T.S. of 935 MPa.

Thus based on Figures 20 and 21, when the brazing time exceeds 360 minutes at 1 200 °C the failure during tensile testing at room temperature occurred in the parent metal.

Figure 22 shows the tensile stress variation at 760 °C as a function of diffusion time at 1 200 °C for butt joints. As can be seen, as the diffusion time is increased, the tensile strength of the joint increased. After diffusion brazing for times of 6,24 and 48 hours at 1 200 °C, tensile testing revealed failure in the joint. However after 72 hours of diffusion brazing at 1 200 °C, fracture occurred in the parent metal during tensile testing. Hence the optimum diffusion brazing cycle from a tensile strength point of view is to hold the joint at 1 200 °C for 72 hours, followed by a solution heat treatment under a hipping pressure of 50 MPa, followed by an aging treatment.

ampio Falled In Parent Metal - Note: Numbered Sample Values Are Each
- Note: Numbered Sample Values Are Each

Figure 20:

MARM 247 Tensile Stress Variation As A Function Of Diffusion Brazing Parameters For Butt Joints With NICRO 150 Filler Metal

*Sampie Falled in Parent Motal

Figure 23 shows the correlation between diffusion time and time to failure during a creep test undertaken at 980 °C and stress of 185 MPa. Prior to creep testing, all diffusion brazed samples were post-solution and aged heat treated. As can be seen the time to failure is only equivalent to the parent metal, after diffusion brazing for 72 hours at 1 200 $^{\circ}$ C. The other important result seen in Figure 23 is that if the parent metal strength was thermally degraded by holding for $24 \rightarrow 72$ hours, then the post solution/hipping and aging heat treatment certainly restores the mechanical strength. This is evident in Figure 23 as the parent metal sample after 24 hours at 1 200 °C followed by the solution/hipping and aging heat treatment, failed at 102 hours when held at 980 °C and 185 MPa whereas the parent metal sample after 72 hours at 1 200 $^{\circ}$ C, followed by the same post heat treatment, failed at 100 hours. This decrease of 2 hours is minimal.

Figure 24 shows the creep rupture properties for joints diffusion brazed at 1 200 $^{\circ}$ C for 72 hours. The creep samples were post-solution heat treated at 1 250 °C for 4 hours at 50 MPa, followed by an age heat treatment at 870 °C for 20 hours. The diffusion brazed joints had nearly an equivalent creep rupture strength to that of the parent metal. Joint efficiencies greater than 95 % was achieved when diffusion brazing at 1 200 \degree C for 72 hours followed by a subsequent heat treatment.

CORRELATION BETWEEN DIFFUSION TIME AND TIME TO FAILURE FOR MAR M 247 DIFFUSION BRAZES

BEFORE CEEFF TESTING ALL BRAZED JOINTS WERE POST HEAT TREATED AT $1250\P^0 C$ FOR 4 HOURS AT 500Pa $+$ $070\P^0 C$ For 20 HOURS

Figure 23:

TOC STRESS 23 *** PARENT METAL** BRAZE JOINT $1²$ 51 $\overline{22}$ $\overline{23}$ 24 $\overline{25}$ 26 $\overline{27}$ $\overline{28}$ $\overline{29}$ $\overline{30}$ $\overline{31}$ $L-M = T(K)[L0G]$ TIME + 20 | X 0.001 -72 HRS AT 1200°C + POST HEAT TREAT FOR 4 HRS AT 1250°C AT 50 MPa + 370 °C FOR 20 HRS

CREEP RUPTURE PROPERTIES OF MAR M 247 DIFFUSION BRAZED JOINTS

Figure 24:

OVERALL DISCUSSION

At 1 125 °C and 1 150 °C regardless of the brazing time (i.e. 20 minutes to 360 minutes), the joint microstructure was undesirable due to the presence of hard, brittle boride phases. All joints brazed for 20 minutes regardless of the brazing temperature had a continuous centre-line phase. This is obviously undesirable for high strength applications as the continuous centre-line boride phase provides an easy path for crack propagation. The increase in time at a specific temperature plays a beneficial role in improving the joint microstructure. At 1 200 °C the Ni-solid solution area and the joint interface disintegrated after brazing for 120 minutes or longer. The diffusion of elements from the parent metal into the joint area and vice versa is greatly enhanced at 1 200 °C for periods longer than 3 hours. No centre-line or boride phase were present in these joints.

With continual increase of time i.e. from 59 to 240 hours at 1 200 °C, the following trend was observed:

- no centre-line or brittle, hard boride phases were present in the joint
- equiaxed grains formed in the joint area
- the volume fraction of the γ ' precipitates in the joint increased
- excessive coarsening of the γ ' precipitates in the parent metal.

Tensile and creep rupture properties are equivalent to the parent metal when diffusion brazing at 1 200 °C for 72 hours. In fact joint efficiencies greater than 95 % was achieved when diffusion brazing at 1 200 °C for 72 hours, followed by a 1 250 °C/4 hours/50 MPa combined solution and hipping heat treatment and a 870 °C/20 hour age heat treatment.

From a mechanical property assessment point of view, it seems that the diffusion of boron into the parent metal did not degrade the properties of the parent metal as all the mechanical property results of the parent metal lied within the scatter range for MAR-M247. Nevertheless, according to Boone et al (1992) an aluminide coating will delay the onset of accelerated oxidation reactions, however the coatings will be depleted more rapidly on the conventional braze repairs than on the control samples prepared from the undoped parent alloy. With this in mind, the effect of this current braze procedure on the cyclic oxidation behaviour of coated MAR-M247 will soon be investigated. Also the effects of re-brazing (re-work) was not really considered in this study. However, it is envisaged that if re-brazing needs to be performed, then the same braze parameters must be used with a post solution and age heat treatment to restore properties in the parent metal and joint if they are degraded via re-brazing. The full effects of rebrazing will also soon be investigated.

The post-solution heat treatment is an important process. It should be realised that single staged aging heat treatments of between 16 to 20 hours at 871 °C are prescribed for MAR-M247, conventionally cast. However, by complete solutioning without incipient melting by soaking at 1 250 °C for 4 hours, the volume fraction of eutectic γ - γ' phase decreases and a refinement of the γ ' size in both the joint area and the parent metal occurred. The 50 MPa pressure applied during the solution treatment is for closure of microporosity present in the joint area and the parent metal.

It has been realised that the total diffusion brazing thermal cycle time is extremely long especially when comparing with the $TLP^{(1)}$, ADH⁽²⁾, LPM⁽³⁾ and SRB⁽⁴⁾ processes, where times in the order of 2-►10 hours (Ellison et al, 1992 and Bell, 1985) could be used for cast Ni-base superalloys. However, these processes are proprietary and licences are required to use these technologies. In the South African context, this could prove very expensive. Thus by using a conventional diffusion brazing filler metal which is available "off the shelf', it was felt that this initially was more economical when comparing to purchasing licences. Also the cost structure e.g. labour time, equipment times, overhead time, in South Africa, is cheaper than compared to Europe and America, hence the long thermal cycle developed here may be uneconomical in Europe and America, however in South Africa it is not.

In this investigation the influence of the joint gap was not studied due to the fact that a 100 μ m filler metal was used. However, it is envisaged that if the filler metal or joint gap was decreased to 50 μ m or even 25 μ m then the diffusion brazing time could be significantly reduced. This is a recommendation for future work. Also based on the fact that the gap width can be controlled by using standard thickness filler metal, a braze replacement insert method shown schematically in Figure 25 could be a viable repair technique for repairing cracks on the nozzle aerofoils. The replacement insert method has been successfully demonstrated by Adam, 1987.

The use of a braze paste or slurry would probably be a more cost effective repair procedure for alloying up individual cracks. However, for this process a halide ion technique is required to clean the oxide off the crack, (Nicoll, 1986) and currently in South Africa no halide ion cleaning facility exists. For this reason the braze replacement insert method is currently being investigated. Also the thermal fatigue properties with respect to this repair scheme will simultaneously investigated.

ERODED OR BRAZING AND REGION PLACE CONTOURING

MACHINE OUT, REPAIR BY GRIND
ERODED OR BRAZING AND INSERT IN
PLACE

Figure 25: Schematic representation of a brazed replacement insert method for repairing cracks on nozzle aerofoils

- (1) TLP is transient liquid phase bonding and is a proprietary process of Pratt and Whitney
- (2) ADH is activated diffusion healing and is a proprietary process of General Electric
- (3) LPM is Liburdi powder metallurgy process and is a proprietary process of Liburdi Engineering Limited
- **(4) SRB** is surface reaction brazing which was developed by Turbine Services

CONCLUSIONS

Using Nicrobraz 150 filler metal to join MAR-M 247, the following conclusions were made:

- Effective and reliable joints were achieved when diffusion brazing at 1 200 °C for 72 hours followed by a 1 250 °C/4 hours/50 MPa combined solution and hipping heat treatment followed by a 870 °C/20 hours age heat treatment.
- Joint efficiencies greater than 95 % was achieved when creep rupture testing. Brazed joint tensile strengths at room temperature are equivalent to that of the parent metal when brazing at 1 200 °C for 360 minutes. However, brazed joint tensile strengths at 760 °C are only equivalent to that of the parent metal when brazing at 1 200 °C for 72 hours followed by the appropriate post braze heat treatment.
- The microstructure of MAR-M 247 diffusion brazed joints resemble the parent metal microstructure closely, with the volume fraction of γ' in the joint being 59 % and in the parent metal 62 %.

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