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THE CONTRIBUTION OF EUTECTIC γ-γ' LIQUID FILM TO THE TMAZ MICROFISSURING IN INERTIA FRICTION WELDED PM RR1000 SUPERALLOY

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Abstract: The microstructure of the base alloy and thermomechanical affected zone (TMAZ) of inertia friction welded powder metallurgy (PM) RR1000 were examined by the use of standard metallographic techniques involving optical, analytical scanning and transmission electron microscopes equipped with energy dispersive spectrometer and an electron probe micro analyser. It was discovered that liquation of grain boundary phases present in the pre-weld alloy resulted in the formation of eutectic type γ-γ' solute rich liquid film predominant in the TMAZ regions which contributed significantly to microfissuring during welding. It was found that apart from the effect of rapid precipitation of γ' particles on the TMAZ cracking as has been widely reported, constitutional liquation of γ' particles was a notable part of liquation process which promoted the susceptibility of TMAZ to microcracking.

Keywords: *Nickel alloys; inertia friction welding; Constitutional liquation; Superalloys; TMAZ microfissuring*

INTRODUCTION

RR1000 is a recently developed PM precipitation-strengthened nickel base superalloy by Rolls Royce Plc, and has been employed in hot sections of gas turbine engines due to its excellent elevated temperature strength and superior hot corrosion resistance. It has been strengthened primarily by precipitation of ordered $L1_2$ intermetallic $Ni_3(Al,Ti)$ γ' phase. This alloy like other precipitation hardened nickel base superalloys that contain substantial amount of Al and Ti (>3wt.%), has been considered very difficult to weld by conventional welding processes due to its high susceptibility to HAZ cracking during welding and strain age cracking post weld heat treatment. Thus, inertia friction welding has been employed in joining the alloy . Cracking during welding of nickel base superalloy has been attributed mostly to large shrinkage stress occurring as a result of rapid precipitation of γ' particles during cooling from welding temperature(Haafkens and Matthey,1982). However, it is known generally that weld cracking results from competition between mechanical driving force for cracking (stress/strain generation) and the material's intrinsic resistance to cracking. It has been reported that liquation which could occur by different mechanisms, is the primary cause of low heat affected zone (HAZ) crack resistance in most austenitic alloys including precipitation hardened Ni base superalloys(Ojo and Chaturvedi,2005). The combined effect of thermally induced welding strain and very low ductility in the alloy due to localized melting of particle at grain boundaries results in HAZ liquation cracking. HAZ or TMAZ liquation is known to occur either by non-equilibrium interface melting below an alloy's solidus or by equilibrium supersolidus melting. Subsolidus HAZ liquation which commonly occurs by constitutional liquation of second phase particles is generally considered more detrimental to crack resistance in that it extends the effective melting range of an alloy and also influences the nature of supersolidus melting by pre-establishing non-equilibrium film at a lower temperature which changes the reaction kinetics during subsequent heating(Bjorneklett *et al*,1998). This process which was first proposed by Pepe and Savage(Pepe and Savage,1967) and has .been observed by different investigators in various alloy system(Romig *et al*, 1988, Pang *et al*,2008 and Ojo *et al*,2006), occurs by a eutectic-type reaction between a second phase particle and the matrix producing a non-equilibrium solute rich film at the particle/matrix interface. It has been discovered that fully austenitic alloys that contain Nb and/or Ti can be highly susceptible to HAZ/TMAZ liquation cracking due to the formation of Nb and/or Ti rich low melting intergranular liquids(Ernst *et al*,1989). It has also been reported recently by Qian and Lippold(Qian and Lippold 2003) that degradation in weldability due to grain boundary liquation in IN 718 resulting primarily from dissolution of Ni_3Nb δ-phase and the associated Nb enrichment of grain boundary has occurred. However, as fundamental as this liquation phenomenon is to HAZ or TMAZ microfissuring, very little information is available about its occurrence in RR1000 superalloy, a recently developed PM superalloy towards the drive in improving gas turbine engine efficiency in modern aircraft engines and power generation system through the increase of Turbine Inlet Temperature (TIT).

The present investigation is aimed at studying grain boundary liquation in the weld TMAZ regions of inertia friction welded RR1000 superalloy in relation to its susceptibility in microcracking.

Experimental procedures

The material used in this work is a new generation nickel base superalloy RR1000, developed
using powder metallurgy (PM) technique. The materials were provided by Rolls Royce PLC, UK. The nickel base alloy of composition (wt%) 15Cr, 16.5Co, 5Mo, 3Al, 3.9Ti, 2Ta, 0.2Hf, 0.02B, 0.05Zr, 0.02C when received was already standard solution heat treated at 1120°C for 4hours and fully aged at 760°C for 8 hours with subsequent air cooling. The As received inertial friction welded sheet samples were of dimensions 20mm X 12mm X 10mm. The samples were sectioned transversely to the weld. The sectioned samples were polished using standard metallographic techniques and were subsequently electrolytically etched in 10% orthophosphoric acid solution at 3.5 V for 3s. TEM foils were prepared by electro polishing in a solution containing 35cc $HClO_4$ in 500cc methanol and 65cc n-butanol at 20V and a temperature ≤ -40°C. The microstructure of the heat treated base alloy and the weld TMAZ were examined and analyzed by the use of optical microscope, field emission gun XL30 scanning electron microscope and Philips CM20 transmission electron microscope, both equipped with energy dispersive X-ray (EDX) spectrometer.

Results and Discussion

A. Microstructure of heat treated base material and TMAZ of the inertia friction welded Part.

The microstructure of the standard solution heat-treated PM base material essentially consists of extensive precipitation of ordered γ' intermetallic phase within the grain and in the intergranular region. This microstructure shows fairly regular coarse, 1–3μm size, primary γ' in the intergranular region and fine (0.1μm) spheroidal dispersion of secondary γ', predominantly in the transgranular region as well as within the intergranular region (Fig. 1). Smaller volume fractions

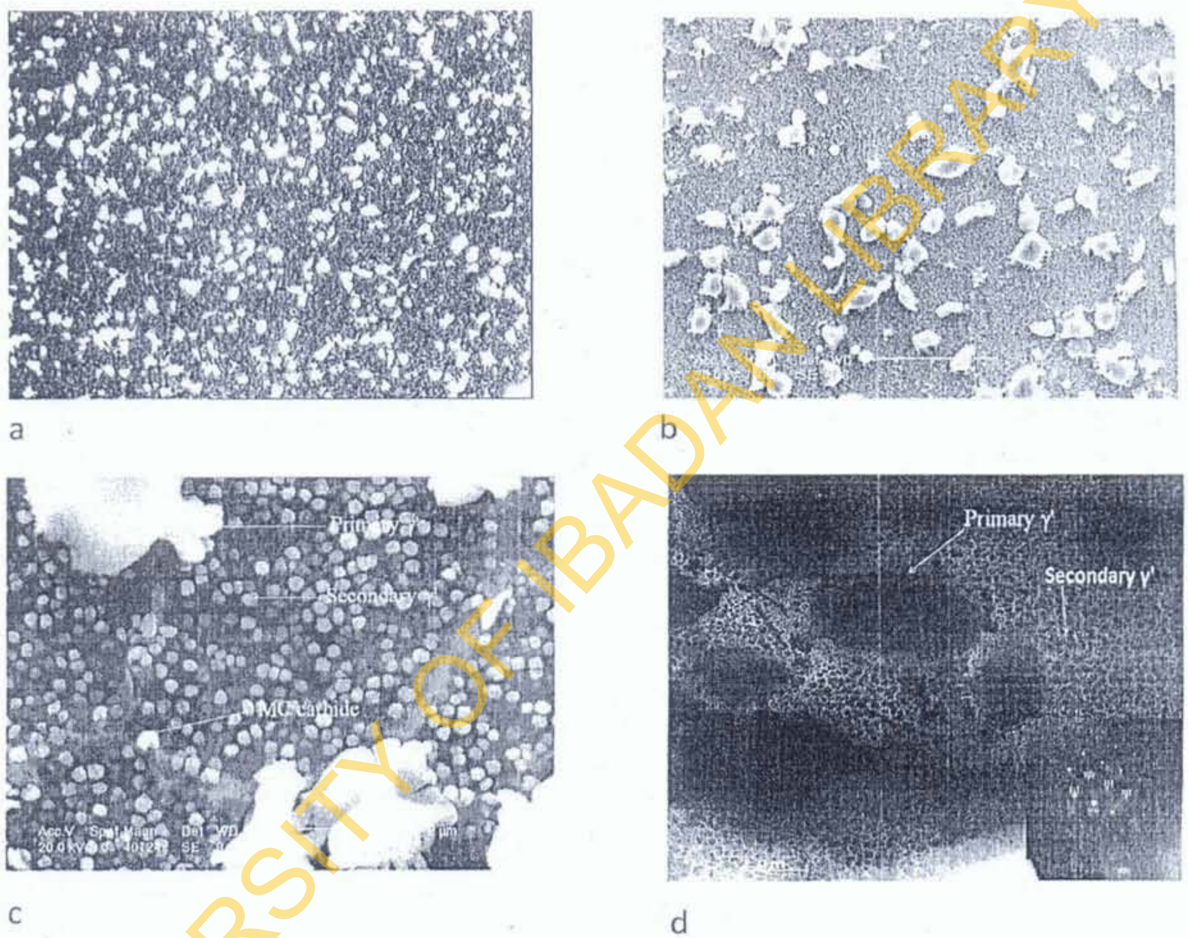
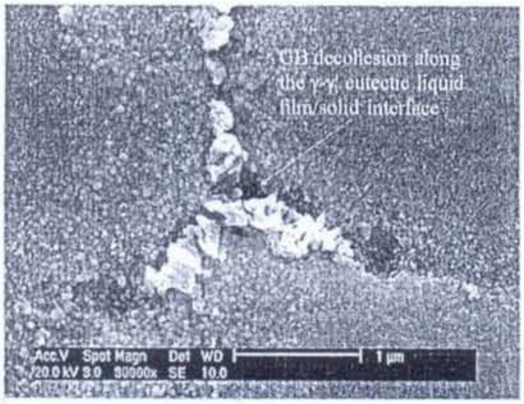


Fig.1 (a) Optical micrograph showing the distribution of primary γ' (b&c) SEM micrograph of γ' and MC carbide in solution heat treated PM RR1000 (d) Bright field transmission electron image with selected area diffraction pattern (SADP) from [$\bar{1}01$] zone axis, showing the distribution of primary and secondary γ' precipitate.

MC type carbide are also present along the intergranular region (Fig 1.). The secondary γ' forms during cooling from the solution treatment temperature. The matrix γ grain is bounded by a ring of large γ' particles due to very rapid growth of these particles that contain higher Al and Ti content, immediately before solutioning. Microcracks were observed in welded samples, with cracking occurring pre-dominantly in the HAZ in the regions slightly removed from the bond line. The cracks displayed a relatively irregular and jagged path typical of liquation cracks, Fig. 2. Closer and careful examination of cracked regions at higher magnification by SEM revealed the existence of re-solidification constituents with eutectic morphology that is characteristic of γ-γ' eutectic which formed at the later stage of solidification in this alloy. The re-solidification constituents, which formed mostly on one side of the cracks as shown in Fig.3 for example, confirm formation of liquid film on the grain boundaries in TMAZ by liquation mechanism. Microfissuring occurred by decohesion across the solid–liquid interfaces under the action of tensile welding stresses generated during cooling.



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b

Fig.2 . Micrographs showing microfissures in (a) 1.5μm from bond line (b) 1μm from bond line

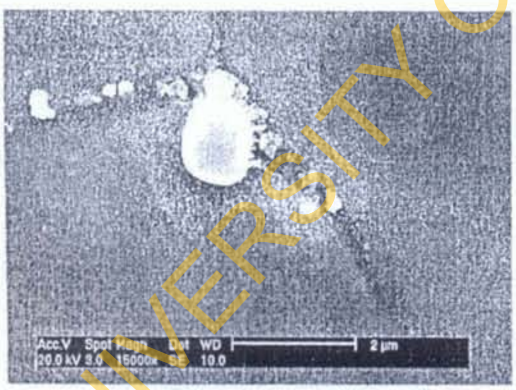

Fig. 3. GB constitutional liquated γ' particle.

B. TMAZ constitutional liquation of phases.

The basis for the TMAZ or HAZ microcracking has been said to exist due to the occurrence of constitutional liquation of an intermetallic compound A_xB_y whose particles persist to at the grain boundary to temperatures equal to or above their eutectic temperature on heating [3]. Inability of the intergranular liquid to accommodate thermal and shrinkage stresses and mechanical constraint during cooling often result to microcracking. Consequently, the susceptibility of an A_xB_y type second phase to constitutional liquation in the weld TMAZ must primarily be linked to its solid state dissolution behavior, as complete dissolution prior to reaching the eutectic temperature will preclude the occurrence of liquation. Several mechanisms have been proposed to explain HAZ or TMAZ grain boundary liquation. The widely accepted being the grain boundary penetration mechanism, where liquid film from liquating particles infiltrates and spreads along the grain boundary regions. The absence of liquating particle on the grain boundary, TMAZ grain boundary liquid penetration requires the simultaneous occurrence of constitutional liquation of intragranular liquating particles and grain boundary migration to the liquating particles [Fig.4].

a b

Fig.4.(a&b) Grain boundary migration to liquated intragranular particle.

However, the penetration of the boundary by the liquid at the particle-matrix interface can either pin the boundary or significantly slows its rate of migration, and thus allows uniform wetting of the boundary by the liquid in some regions (Fig2a).

The present work has also shown that aside the super solvus melting which is expected to occur in all weldments due to heating above the equilibrium solidus temperature, constitutional liquation of MC-type carbides present in the pre-weld alloy were observed to contribute to the liquation process and consequently affecting the TMAZ microcracking (Fig.5). Titanium rich particle was also observed as a part of the re-solidified product along some crack regions (Fig.6). Liquid titanium has been found to be a melting point depressant, thus the presence of this Ti rich particle could aggravate the liquation of grain boundary particle.

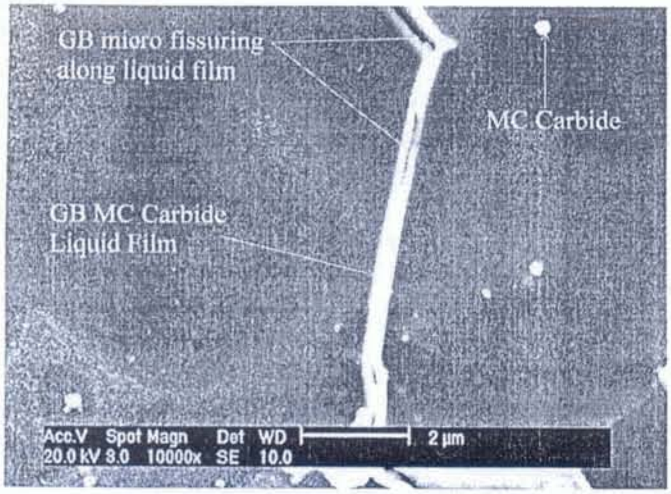

Fig.5. Infiltration of MC liquid film along the GB.

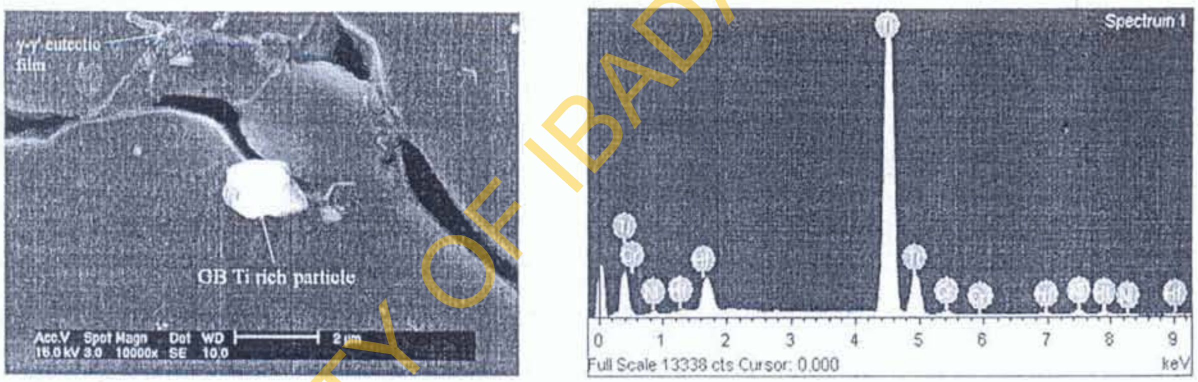

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Fig.6(a) Micrograph showing GB Ti rich particle. (b) EDX spectrum of the Ti rich precipitate.

An important part in this present investigation is the high volume fraction of γ' in this recently developed superalloy, RR1000 could have a significant deleterious effect on the performance of the weld in service due to the presence of the increased γ' forming elements. This is in addition to the widely reported rapid precipitation of these particles during cooling from weld temperature that induces significant amount of thermal stress.

The reason for the survival of these particles at such peak welding temperature, which is beyond their solvus temperature, could be due to the limited integrated time available for homogenization by diffusion process during rapid heating of inertial friction welding.

The temperature of γ-γ' eutectic reaction in nickel base superalloys has been reported to occur between the range of 1180-1198°C(Rosenthal and West, 1999 and Ojo et al,2004) which is always below the equilibrium solidus temperature. Dissolution behaviour of γ' precipitates is expected to deviate from equilibrium due to rapid thermal cycling involved during welding. An attempt has been made to model particle dissolution under rapid heating condition by an analytical technique as well as through the additivity and isokinetic approach(Bjorneklett *et al*,1998). The results of the two methods, which were found to be in good agreement with numerical dissolution model and experimental results, show that the degree of particle dissolution depends on interplay between the heating rate and the initial particle size. The solid state dissolution of the γ' phase in Astroloy superalloy at equilibrium and under rapid heating was studied by Soucail and Bienvenu(Soucail and Bienvenu,1996) in separate work. Their results, which are in agreement with those of Bjorneklett *et al* (1998) showed that there is a significant deviation from equilibrium under rapid heating condition, in that the temperature of complete solid state dissolution increased with increasing heating rate and this deviation is dependent on the initial particle size. This increase in complete dissolution temperature was found to be more pronounced with increase in particle size. An increase of about 120°C in complete dissolution temperature was reported for γ' precipitates with initial size of 0.8μm under a heating rate of 8°C/s. In inertia friction welding process, like the one used in the present work, typical heating rate normally exceeds 150°C/s and as such, variations in γ' dissolution behavior of γ' particles can be expected to depend on the particle's location and size, with the possibility of some coarse particles remaining undissolved above 1200°C. The eutectic reaction between such incompletely dissolved particle and the solute rich γ matrix surrounding such particle can result in constitutional liquation and subsequent penetration of the liquid along the grain boundary. Constitutional liquation of both intergranular and intragranular γ' were observed in the TMAZ of RR1000 superalloy. The degree of liquation increased with decrease distance. To avoid argument that liquation of intergranular γ' particles cannot be used to conclude the occurrence of constitutional liquation of the precipitate, knowing that other liquation mechanisms may also be operative at grain boundaries, such as constitutional liquation of MC type carbides as illustrated in Figure 5 and possibly liquation due to segregation of low melting point depressing elements like titanium (Fig.6). Consequently intragranular particles located up to 10μm away from HAZ grain boundaries and distinctly separated from other liquating phases were closely examined. Evidence of γ/γ' interface liquation was observed not only along the grain boundaries but more importantly within the grains of the HAZ (Figs.4&7). The solute rich liquid pool adjacent to liquating particles is expected to commence solidification first as gamma and then on reaching the eutectic temperature transform to γ-γ' eutectic product. On further cooling, γ' could precipitate out of the newly formed supersaturated γ phase resulting in a re-solidified region consisting of coarser γ' precipitate versus adjacent unmelted matrix and fine γ-γ' eutectic protruding into the last area to solidify. The volume and composition of liquid present at the peak temperature is a function of the microstructure that forms. These two types of re-solidified morphology were observed in this work with the intragranular particles having fine re-solidified structure. High magnification SEM image suggests that some of these re-solidified regions contain fine γ-γ' eutectic which formed at the terminal stage of the solidification process. This suggestion was further supported in instances where a complete liquation of the intragranular precipitates occurred (Fig.8). Fine γ-γ' eutectic colonies were oriented with their ''crown'' region protruding into the last liquid to solidify, which is the typical mode of γ-γ' eutectic formation(Zhu *et al*,1988). An additional indication of constitutional liquation is the observation of what appears to be voids or cavities in the immediate vicinity of extensively liquated intraganular particles (Fig. 9). It is known that voids and cavities often form in melted and resolidified regions in superalloys owing to the expansion and contraction accompanying solidification. It has been reported that atom probe field ion microscopy (APFIM) study of nickel base superalloys showed that there was no boronor titanium segregation to the interfaces of intragranular γ' particles(Blavette *et al*,1996). Thus, the occurrence of a liquid film surrounding the intragranular γ' precipitates which are away from other liquating phases can only be reasonably attributed to constitutional liquation of the intermetallic particles(Pepe and Savage,1967). It was observed that the closer these particles were to the fusion line, that is increased peak temperature, the more pronounced the liquation. This indicates that the coarse particles that did not completely dissolve before reaching the eutectic temperature, constitutionally liquated, with the extent of liquation increasing with the increase in peak temperature experienced in the TMAZ.

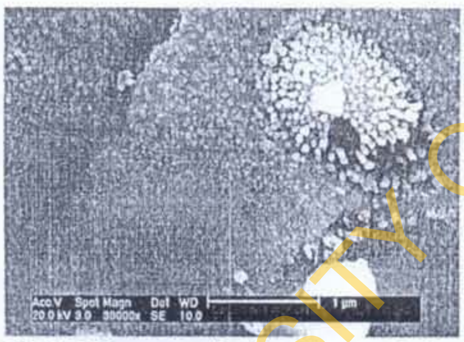

Fig.7 Micrograph showing an intragranular liquated γ' particle

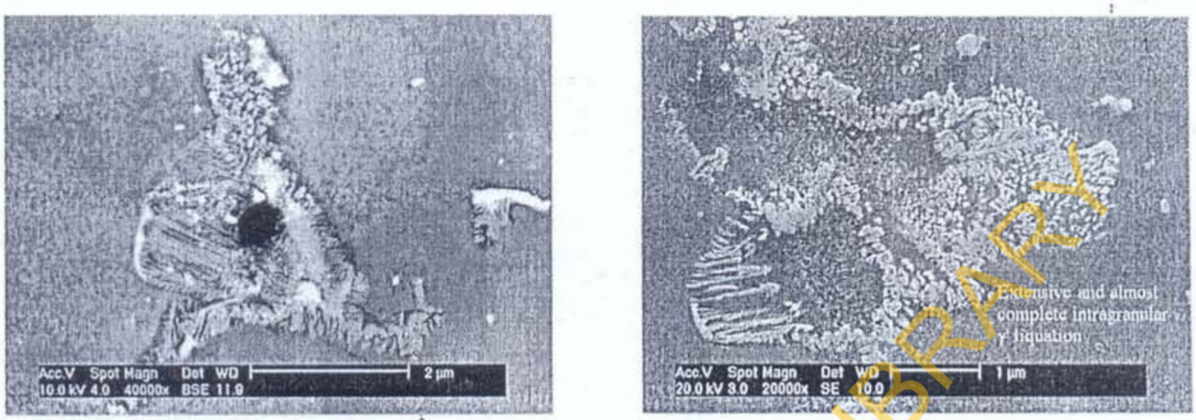

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Fig.8 (a) Back scattered image mode showing crack formation due to eutectic γ-γ' solid-liquid interface decohesion along GB constitutional liquation of intergranular (b) morphology of an extensive intergranular γ-γ' eutectic product.

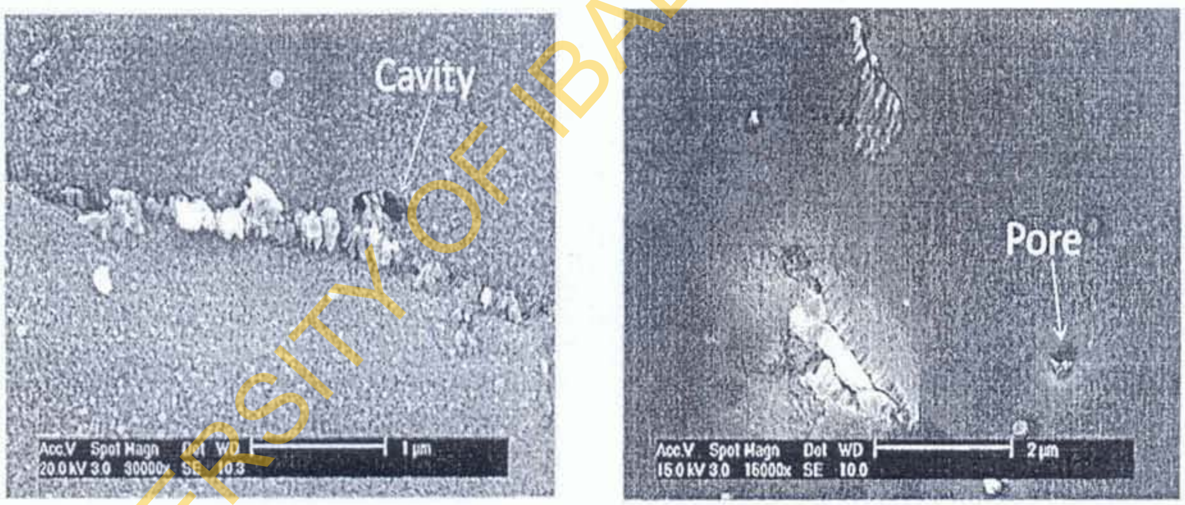

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Fig.9 (a) Micro void in the vicinity of near GB precipitate (b) Micro pores showing evidence of intragranular liquation

C. Effect of constitutional liquation of γ' particles on the mode of re-solidification of intergranular liquid film and its suscepibilty to microcracking.

γ' particle liquation has been significant due to its considerable contribution to intergranular liquid film thickness which would reduce the stress required to cause cracking by decohesion of solid-liquid interface (Fig.8a) as illustrated by the equation; $\sigma = 2\gamma_L/h$. (where σ is the tensile stress required to overcome surface tension γ_L on a boundary containing liquid film of thickness h). This implies that any parameter which contributes to an increase in the grain boundary film thickness h, would reduce the crack resistance. However, mere occurrence of liquation is not sufficient to produce a crack susceptible microstructure. Susceptibility to cracking depends on penetration and wetting of grain boundary liquid film thickness and its stability to temperatures at which sufficient thermal and mechanical stresses are generated on cooling. Grain boundary wetting is enhanced if the solid–liquid interfacial energy is small compared to the grain boundary energy. Considering that the metastable liquid produced by constitutional liquation always reacts with the solid through solute back diffusion, the non-equilibrium solid–liquid interface energy is very low(Aksay *et al*,1974), and as such extensive grain boundary penetration and wetting by film produced by constitutional liquation of γ' particles is expected. This was observed in all the samples with significant penetration and spreading of the film along the grain boundary (Fig.2, 4&5), even to the lower temperature subsolidus region of the TMAZ. Interaction of the film with grain boundary microsegregated titanium rich particle could also aid the wetting behavior, as titanium rich phase was observed as part of the re-solidification constituents along some cracked grain boundaries (Fig.6). This is because on cooling, the low diffusivity of Ti in the surrounding austenite matrix and its low partition coefficient (0.6) could enable the existing liquid to persist to a much lower eutectic temperature, thereby increasing the local effective solidification range which would reduce the crack resistance(Ojo et al,2004).

Attention should be focussed on the fact that γ' precipitates are an essential strengthening phase which are considered better to be in coarsened form in pre-weld material to induce enhanced ductility in the material for relieving welding strains. The confirmation of constitutional liquation of γ' precipitates in this alloy is considered crucial and significant. Its high volume fraction and the lower eutectic temperature relative to the alloy's equilibrium solidus is therefore a point of concern. This communication has therefore been shown that in addition to the effect of rapid on-cooling precipitation of γ' on TMAZ microcracking, the occurrence of constitutional liquation of γ' precipitate could also cause a deleterious effect on TMAZ microcracking in the recently developed RR1000 superalloy.

CONCLUSIONS

This investigation has shown that extensive grain boundary liquation occurs in the TMAZ of inertia friction welded RR1000 superalloy. The major strengthening phase in the alloy, γ' has been found to contribute significantly to the liquation phenomenon and the attendant cracking of the TMAZ. MC type carbides in the pre -welded alloy and microsegregated titanium particle formed during welding have also contributed to the microcracking of the TMAZ through liquation process.

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