# TMAZ MICROCRACKING IN INERTIA FRICTION WELDING OF PM RR1000 SUPERALLOY: CONCOMITANT EFFECT OF CONSTITUTIONAL LIQUATING PARTICLES

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## ABSTRACT

*The effect of constitutional liquation of strengthening particles in PM RR1000 superalloy in response to inertia friction welding process was investigated. The thermomechanical affected zone (TMAZ) microstructures were simulated using Gleeble$^{R}$ thermomechanical simulation system. The microstructural examination of the simulated TMAZs and those from the actual welded specimens showed the occurrence of extensive grain boundary liquation of the main strengthening phase $\gamma'$ and MC type carbide, and consequently attendant microcracking in the TMAZ of the alloy. The existence of these particles in the temperature range well above their solvus temperatures orchestrated the eutectic type reaction leading to their constitutional liquation and subsequent cracking of the weldment.*

**Keywords**: *Nickel alloys; Welding; Constitutional liquation; Superalloys; TMAZ microcracking*

## 1.0 INTRODUCTION

RR1000 is a PM precipitation-strengthened nickel base superalloy which 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 $Ni_3(Al,Ti)$ $\gamma'$ phase. RR1000, like other precipitation hardened nickel base superalloys that contain substantial amount of Al and Ti (>3wt.%), has been considered very difficult to weld due to its high susceptibility to HAZ cracking during welding and post weld heat treatment by strain age cracking Ojo et al. (2008). Cracking during welding of nickel base super alloy has been attributed mostly to large shrinkage stress occurring as a result of rapid precipitation of $\gamma'$ particles during cooling from welding temperature. 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 Haafkens and Mathey, (1982). It has been discovered 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 at grain boundaries results in HAZ liquation cracking. HAZ and TMAZ liquation are 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 Ojo et al, (2005). This phenomenon which was first proposed by Pepe and Savage (1967) and has been observed by different investigators in various alloy system (Romig et al, 1988 and Ojo et al, 2006), occurs by a eutectic-type reaction between a second phase particle and the matrix producing a nonequilibrium solute rich film at the particle/matrix interface. Research work has also shown that fully austenitic alloys that contain Nb and/or Ti can be highly susceptible to HAZ and TMAZ liquation cracking due to the formation of Nb and/or Ti rich low melting intergranular liquids Ernst et al, (2003). It has also been reported recently by 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. Nevertheless, as fundamental as this liquation phenomenon is to HAZ and TMAZ microfissuring, very little information is available about its occurrence in RR1000 superalloy. The above results, alongside with the fact that Al and Ti (especially Ti, which also segregate into liquid in nickel base alloy) are melting point depressants, suggest that apart from the rapid precipitation effect of $\gamma'$ phase on TMAZ microfissuring, these $\gamma'$ elements could also be contributing to high TMAZ microfissuring susceptibility in $\gamma'$ precipitation-hardened alloys like RR1000 in other ways.

In the present communication, constitutional liquation of MC carbides, and coarse $\gamma'$ precipitate was observed to have contributed to the TMAZ liquation and its attendant microfissuring. Constitutional liquation of carbides, borides and sulfides has been reasonably well discussed in other superalloy weldments Ojo et al, 2005, Pepe, et al, 1967, and Ojo et al, 2006), but constitutional liquation of $\gamma'$ precipitates in RR1000 superalloy system, a recently PM developed 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) has not been reported in literature. Clear evidence of constitutional liquation of $\gamma'$ phase was observed in this work and was found to be closely associated with the TMAZ microfissuring. The presence of significant amount of $\gamma'$ in the pre-weld solution treated condition (48%) and the fact that it is the prime strengthening phase of the alloy means that liquation of $\gamma'$ in this alloy would have very serious effect on its weldability. This communication reports the detection of constitutional liquation of $\gamma'$ precipitate in inertial friction welded RR1000 superalloy and discusses its contribution to TMAZ microfissuring.

## 2.0 EXPERIMENTAL PROCEDURE

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 themomechanical affected zone microstructure was simulated by a Gleeble thermomechanical simulation system. The cylindrical specimens used for the Gleeble simulation were 8mm diameter and 7.96mm length. Simulated temperatures were 1175 and 1225°C, heating at a rate of 10°C/s and held for 1s at both temperatures followed by water quenching. A chromel-alumel thermocouple was spot welded to the specimens at the midsection to monitor temperature. The specimens were water quenched (i.e. cooling at a rate of about 130°C/sec) subsequent to the rapid heating in order to preserve, as much as possible, the microstructural changes that occurred at the simulation temperatures. Inertial friction welded samples were sectioned transversely to the weld, and the Gleeble simulated samples were sectioned at the location of the spot welded thermocouple. 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.

## 3.0 RESULTS AND DISCUSSION

**Microstructure of heat treated base material, TMAZ of the inertia friction welded part and simulated samples.**

The microstructures of the base material RR1000 showing the distribution of γ' precipitates in the solution treated material matrix are presented in Figure 1. Figure2 shows microfissuring in the as welded material and in the TMAZ Gleeble simulated specimen. Figure 3 shows constitutional liquated γ' particle in the microstructure of the TMAZ.

The microstructure of the heat-treated PM base material was seen to consist essentially 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' and very fine tertiary γ' (5-30nm), predominantly in the transgranular region as well as within the intergranular region (Figure 1).

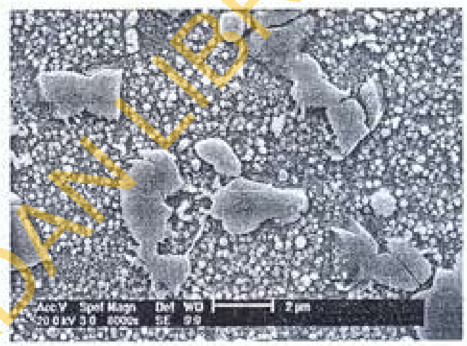
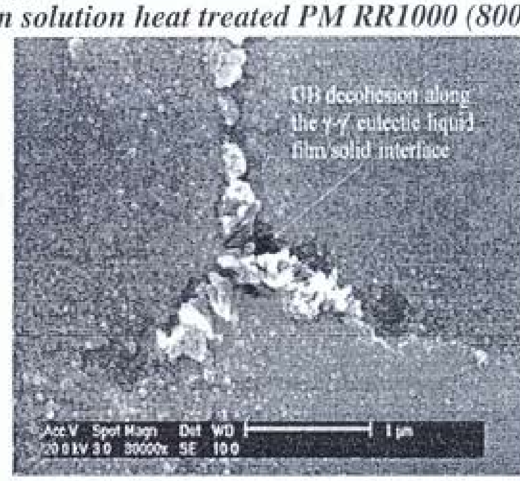


*Figure 1: SEM micrograph of γ' precipitates in solution heat treated PM RR1000 (8000x)*

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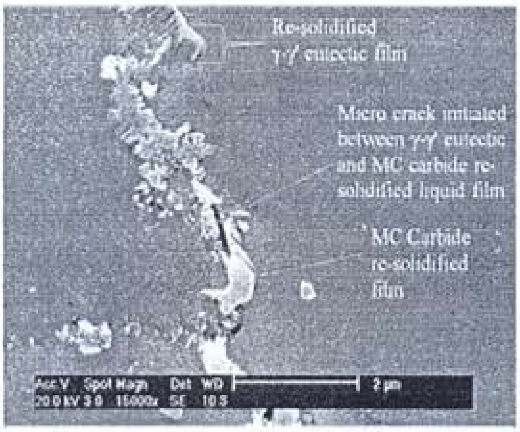


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***Figure 2: Micrographs showing microfissures in (a) as weld condition(30000x) (b)TMAZ simulation at 1175°C(15000x).***

Smaller volume fractions of MC type carbide are also present along the intergranular region (Figure 1). The secondary γ' formed 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 of similar morphology were observed in welded samples and simulated samples, with cracking occurring predominantly 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, Figure 2. Closer and careful examination of cracked regions at higher magnification by SEM (Figure 3) 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 Figure 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.

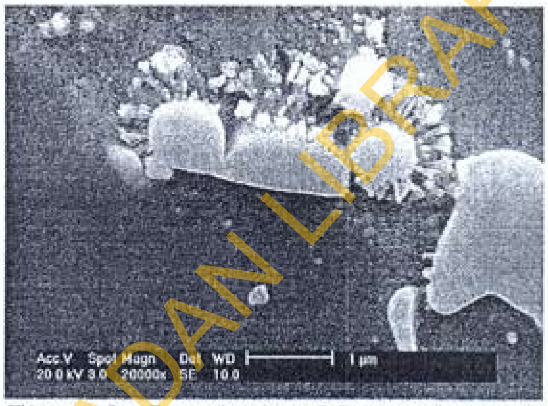


***Figure 3: Constitutional liquated γ' particle in TMAZ simulation (20000x)***

**Liquation phenomenon in γ' precipitate particles**

Figure 4 is the micrograph showing formation of MC type carbide liquid film along the grain boundry(GB). Figure 5(a) is a micrograph showing GB Ti rich particle. While Figure 5(b)is an EDX spectrum of the Ti rich precipitate. Fig.6 is a micrograph showing intragranular liquated γ' precipitate while Figure 7 is a micrograph illustrating extensive intragranular liquation of γ' precipitate of the TMAZ simulated sample at 1225°C. Figure 8(a) is a micrograph showing grain boundary liquation of γ-γ' eutectic product and Figure 8(b) shows liquated of intragranular γ' particles

The fundamental requirement for the occurrence of constitutional liquation of an intermetallic compound $A_xB_y$ in an alloy is the existence of $A_xB_y$ particles at temperatures equal to or above their eutectic temperature on heating Pepe et al, (1967). Consequently, the susceptibility of an $A_xB_y$ type second phase to constitutional liquation in the weld TMAZ must primarily be related to its solid state dissolution behavior, as complete dissolution prior to reaching the eutectic temperature will preclude the occurrence of liquation. The terminal eutectic temperature in Inconel 718 above which constitutional liquation of $A_2B$-type Laves phase and NbC particles were observed was found to correspond to the terminal reaction peak temperature during thermal analysis (7).

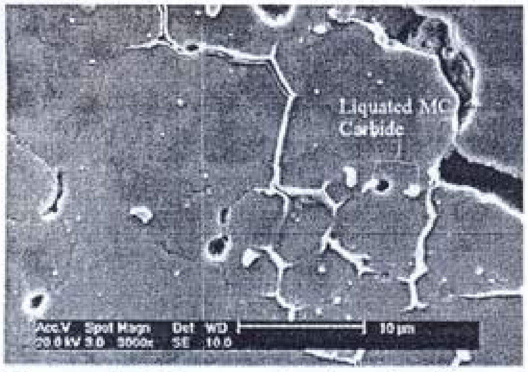



*Figure 4: Micrograph showing formation of MC liquid film along the GB(3000X)*

It has been reported that the temperature of γ-γ' eutectic reaction in nickel base superalloys occurs between the range of 1180-1198°C (Bjorneklett et al, 1998 and Soucail et al, 1996) which is always below the equilibrium solidus temperature (Rosenthal et al, 1999 and Sallemark et al, 1975). Dissolution behavior 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 (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 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 resulting in their constitutional liquation. 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 4 and possibly liquation due to segregation of low melting point depressing elements like titanium (Figure 5). 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 TMAZ (Figure 6).

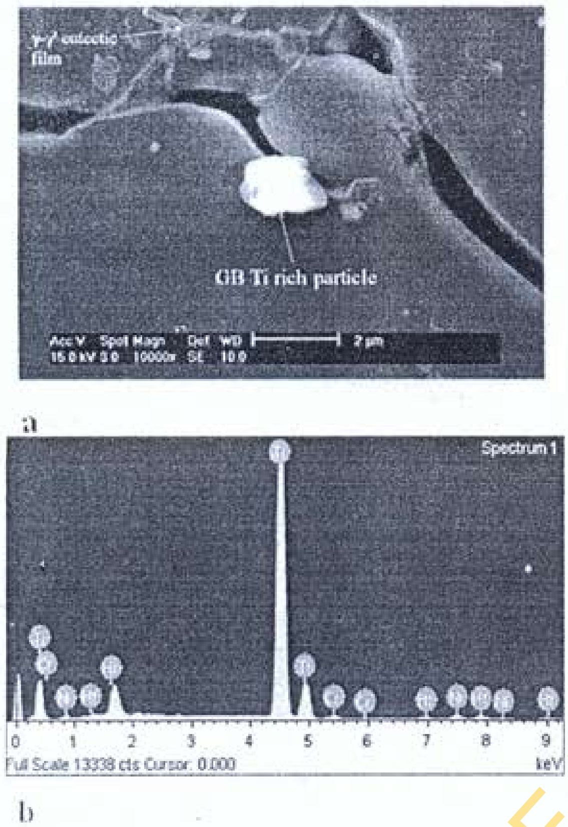



*Figure 5 (a) Micrograph showing GB Ti rich particle(10000X) (b) EDX spectrum of the Ti rich precipitate.*

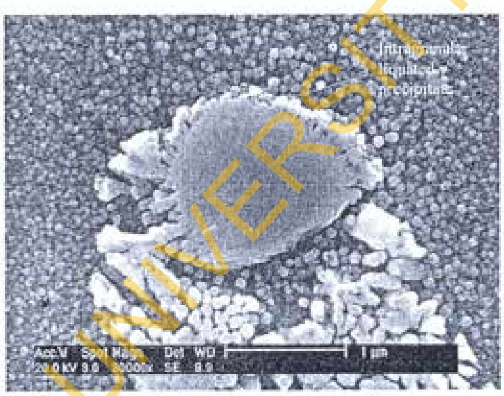



*Figure 6: Micrograph showing intragranular liquated precipitate(30000x)*

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 super-saturated 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 (Figure 7). This suggestion was further supported in instances where a complete liquation of the intragranular precipitates occurred. 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 (Figure 4). 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 boron or titanium segregation to the interfaces of intragranular γ' particles Blavette(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 et al, (1967). It was observed that the closer these particles were to the weld line, that is increased peak temperature, the more pronounced the liquation. This indicated 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.

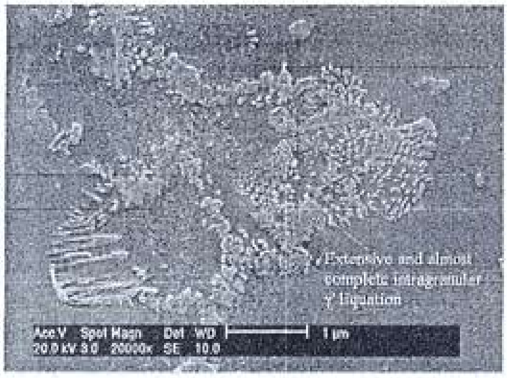
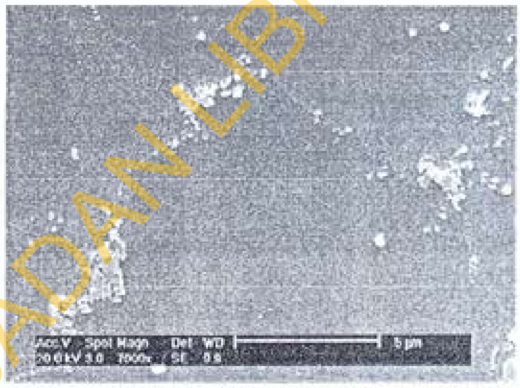



***Figure 7: Micrograph illustrating extensive intragranular liquation of γ' precipitate of the TMAZ simulated sample at 1225°C(20000x)***

## Effect of constitutional liquation of γ' particles

The 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 (Figures 2 & 4), even to the lower temperature subsolidus region of the TMAZ. Interaction of the film with grain boundary segregated surface active elements like boron/titanium could also aid the wetting behavior, as titanium rich phase was observed as part of the re-solidification constituents along some cracked grain boundaries (Figure 5).

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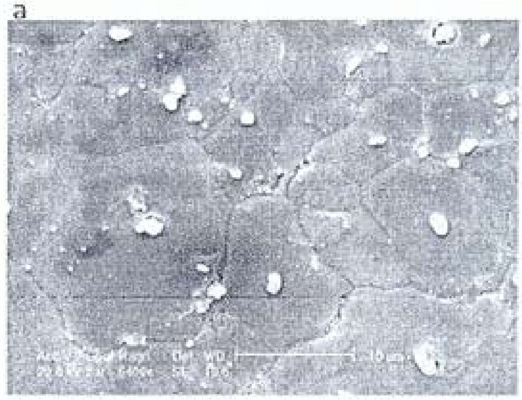

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***Figure 8: (a) Micrograph showing grain boundary liquation of γ-γ' eutectic product (7000x) (b) Liquation of intragranular γ' Particles (6400x)***

Furthermore, as can be seen from the following expression Miller et al, (1967), $\sigma = 2\gamma_L/h$. (where $\sigma$ is the tensile stress required to overcome surface tension $\gamma_L$ on a boundary containing liquid film of thickness h), any parameter which contributes to an increase in the grain boundary film thickness h, would reduce the crack resistance. The high volume fraction of liquating γ' is a factor that could increase film thickness and thus make the PM RR1000 alloy more susceptible to weld cracking. Liquid film penetration from constitutionally liquated γ' particles in adjacent grains and close to the grain boundaries were found to contribute to intergranular liquation (Figure 8). In terms of film stability to lower temperatures, this can be assessed by considering subsolidus portion of the TMAZ. The result of Gleeble thermal simulation showed that considerable γ' liquation is occurring as low as 1175 °C. In this HAZ region, complete liquation of coarse particles may not be realized during the time the specimen remains at peak temperature during welding. 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 the 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 due to it's inevitable presence as the main strengthening phase, high volume fraction and the lower eutectic temperature relative to the alloy's equilibrium solidus. It is therefore observed that in addition to the effect of rapid on-cooling precipitation of γ' on TMAZ microfissuring, the occurrence of constitutional liquation of γ' precipitate could also cause the deleterious TMAZ microfissuring in RR1000 superalloy and other precipitation strengthened nickel base superalloys.

## 4.0 CONCLUSIONS

1. Constitutional liquation of both intragranular and intergranular γ' precipitates was found to be contributing significantly to the TMA liquation in inertia friction welded RR1000 superalloy.

2. Resolidified constituents were observed Along cracked grain boundaries in the HAZ of welded PM RR1000 superalloy and it is concluded that one of the factors responsible for TMAZ microfissuring in this alloy is grain boundary liquation.

3. Liquid films from liquated γ' and MC carbide precipitates were observed to exhibit extensive penetration and wetting of grain boundaries even to the lower temperature subsolidus region of the TMAZ.

3. Constitutional liquation of γ' precipitates could as well be a contributing factor to the high TMAZ crack susceptibility of other precipitation hardened nickel base superalloys with high volume fraction of γ' precipitate particles.

## 5.0 REFERENCES

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