

Effect of deformation mode on hot ductility of a γ' precipitation strengthened nickel-base superalloy

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ABSTRACT

Disparity in hot ductility of a γ' precipitation strengthened nickel-base superalloy, IN 738 LC, subjected to non-equilibrium heating and compressive and tensile stresses, was investigated. The alloy, which shows considerable hot ductility at temperatures ranging from 1160 °C to 1250 °C under compressive loading, exhibits zero ductility under tensile loading within this temperature range. The difference is attributed to the fact that while compressive loading permits plastic deformation in spite of non-equilibrium liquid phase dissolution of γ' precipitates in the alloy, the occurrence of the liquation reaction results in inhibition of plastic deformation under tensile loading. Accordingly, while grain refinement through strain-induced dynamic recrystallization occurred under compressive loading, within the same temperature range, the formation of new grains was prevented under tensile loading. This behavior is crucial during high temperature processing of γ' precipitation strengthened nickel-base superalloys.

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1. Introduction

Manufacturing of components for applications in the high temperature operating environment of aero and land-based gas turbine engines requires the use of materials with excellent high temperature mechanical properties and reliable hot corrosion resistance. Nickel-base superalloys are usually used for hot-section gas turbine applications. The high temperature performance of nickel-base superalloys can be attributed to a combination of solid solution strengthening by a careful selection of alloy chemistry, precipitation strengthening of the matrix by γ' and/or γ'' phases, and improvements in grain boundary characteristics by the formation of various carbide phases. Cast nickel-base superalloys that are precipitation strengthened by γ' precipitates, such as IN 738 LC, are particularly suited for higher operating temperatures due to better microstructural stability.

Joining and forming processes are important in the manufacturing of gas turbine components. Recent developments in the joining of high temperature materials have led to the use of friction joining processes, including friction stir, linear friction, etc., which have produced excellent joints in materials that are very difficult to join by other conventional methods [1–4]. Also, forming processes, such as forging, extrusion and rolling, remain critically important in the manufacturing of gas turbine components due to the possibility of

achieving high production volumes and various shapes [5,6]. Friction joining and forming involve two major events. Firstly, the materials are rapidly heated (non-equilibrium heating) to the processing temperature, which usually results in drastic changes in microstructure. Secondly, the materials undergo significant plastic deformation under externally imposed stresses. Studies have shown that plastic deformation and hot workability of γ' precipitation strengthened nickel-base superalloys depend on the dissolution behavior of the precipitates at processing temperatures [7,8]. Precipitation strengthened nickel-base superalloys, such as IN 738 LC, are usually hot and difficult to work due to their high flow stresses. This necessitates an increase in hot working temperatures, where the precipitates essentially dissolve in order to enhance workability.

One of the phenomena controlling the flow stress and the rate of crack propagation in hot-worked materials is dynamic recrystallization, which occurs in several alloys, including precipitation strengthened nickel-base superalloys [7]. During hot working deformation, several metals tend to exhibit a microstructure consisting of dislocation sub-boundaries similar to the structure obtained after softening of cold-worked materials by annealing but without recrystallization [7]. This is referred to as dynamic recovery and has been observed in different materials [7,9]. High strains and strain rates increase the density of dislocations at the sub-boundaries and cause the dislocations to become more tangled [10]. Due to greater misorientation between the sub-grains, created by the network of dislocations, new grains eventually nucleate, lowering the flow stress and resulting in a steady-state condition where deformation, recovery

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and recrystallization occur at the hot working temperature. The occurrence of such dynamic recrystallization has been observed in several materials [6,7,11]. Dynamic recrystallization and other phenomena associated with hot working of precipitation strengthened nickel-base superalloys depend significantly on the behavior of the strengthening γ' precipitates. Therefore, the behavior γ' precipitates during high temperature processing is crucial to the material's overall response to processing. The objective of this work was to study plastic deformation behavior of γ' precipitation strengthened nickel-base IN 738 LC superalloy subjected to non-equilibrium heating and compressive and tensile stresses. The results are reported in this communication.

2. Materials and experimental procedure

Cast IN 738 LC superalloy, with a nominal composition of (wt pct) 0.11C, 15.84Cr, 8.5Co, 2.48W, 1.88Mo, 0.92Nb, 0.07Fe, 3.46Al, 3.47Ti, 1.69Ta, 0.04Zr, 0.012B and balance nickel, was received in the form of plates of $240 \times 60 \times 15$ mm³ dimension. The plates were given the standard solution heat treatment (SHT) at 1120 °C for 2 h, followed by air cooling. The ductility behavior of the alloy was studied by using a Gleeble 1500-D Thermo-Mechanical Simulation System. Cylindrical Gleeble specimens of 6 mm diameter and 115 mm length were used for the tension tests, while specimens of 6 mm diameter and 10 mm length were used for the compression tests. Gleeble simulations were performed by rapidly heating the specimens at heating rates from 111 °C s⁻¹ to 150 °C s⁻¹ and to temperatures ranging from 1100 °C to 1250 °C, while tensile or compressive stresses were applied at the peak temperatures accordingly. Certain specimens were simulated to study the effect of thermal cycle alone, without imposed stress. These were heated to the peak temperatures and held for specific holding times ranging from 0.5 s to 10.5 s, then air cooled. The temperatures of the specimens were controlled and measured during Gleeble testing by spot-welding chromel–alumel thermocouples at the longitudinal axial center of each specimen. Ductility was evaluated in terms of percent change in cross section at the midsection of each sample by measuring the diameter of fractured tensile test pieces and by measuring the diameter of transversely sectioned compression test pieces using a low magnification Nikon optical microscope. Solution heat treated and Gleeble-tested specimens (sectioned at the location of the spot-welded thermocouples) were prepared by standard metallographic techniques and etched electrolytically in 12 mL H₃PO₄+40 mL HNO₃+48 mL H₂SO₄ solution at 6 V for 5 s for microstructural analysis. Selected samples were chemically etched by dipping in Kallings reagent for 30 s. Microstructures of the materials were examined and analyzed by using a ZEISS Axiovert 25 inverted reflected-light optical microscope equipped with a CLEMEX vision 3.0 image analyzer (Clemex Technologies Inc., Longueuil, Canada) and a JSM 5900 scanning electron microscope (SEM) equipped with an Oxford (Oxford Instruments, Oxford, United Kingdom) ultrathin window energy-dispersive spectrometer (EDS) and Inca analyzing software. The grain structure of a compressed specimen was studied by carrying out electron backscatter diffraction (EBSD)-based orientation analysis using an HKL Nordlys EBSD detector (developed by Oxford Instruments), which was attached to a Philips XL 30 Scanning Electron Microscope and equipped with Oxford Instrument HKL Technology Channel 5 suite of programs. Grain mapping was carried out at a step size of 1.5 μ m.

3. Results

3.1. Microstructure of the solution heat treated (SHT) material

The low magnification light-optical image of the solution heat-treated (SHT) material (Fig. 1a) reveals the grain structure of the

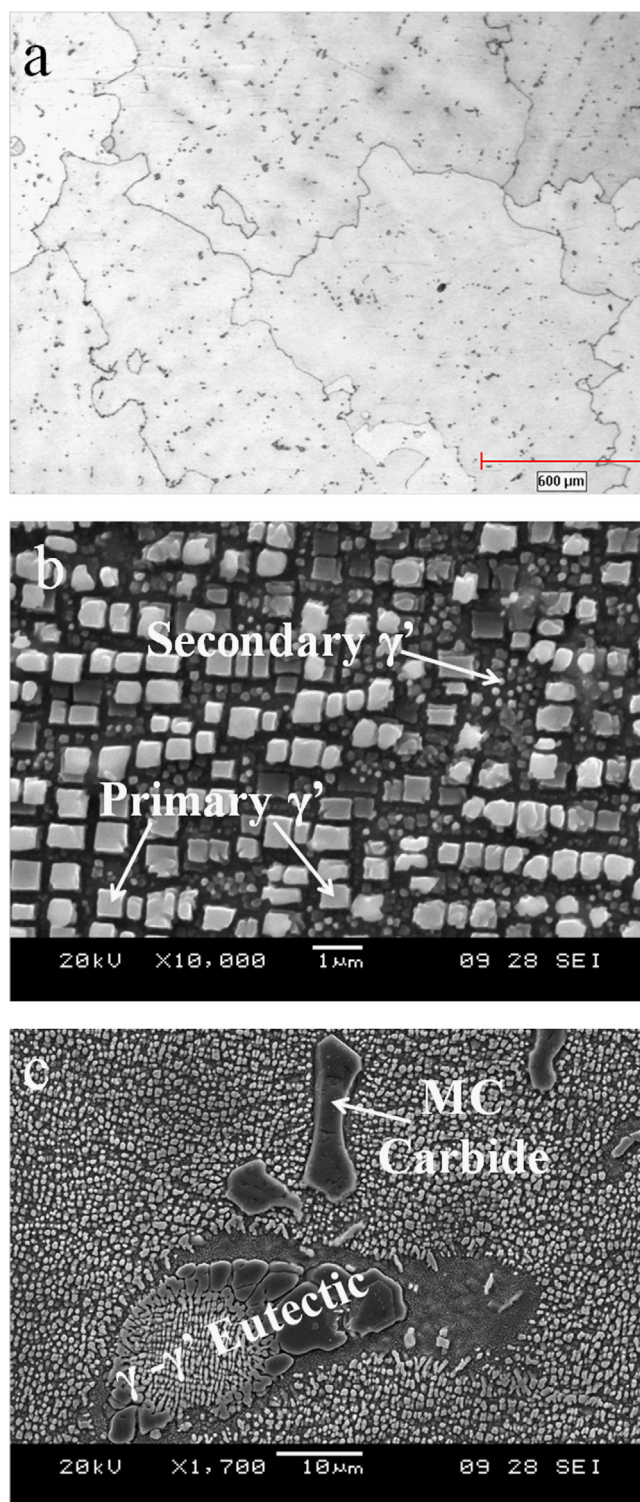


Fig. 1. (a) Low magnification light-optical image of the solution heat-treated IN 738 LC superalloy showing the grain structure, and SEM micrographs showing (b) primary and secondary γ' precipitates, and (c) γ – γ' eutectic and MC carbide.

material before thermo-mechanical processing. Fig. 1(b and c) shows SEM micrographs of the SHT material. The microstructure consisted of coarse primary γ' precipitates with sizes ranging from about 0.4–0.8 μ m, fine spherical secondary γ' precipitates of about 0.1 μ m in diameter, and solidification products that formed during casting of the plates, namely MC carbides and γ – γ' eutectics. These microconstituents have been previously reported in SHT IN 738 LC superalloy [12,13].

3.2. Gleeble hot ductility testing

Gleeble hot ductility testing is usually used to evaluate the deformation behavior of materials, which reflects the ability of the materials to accommodate externally imposed stresses [14]. Gleeble testing could be either tensile or compressive testing. Gleeble hot tensile and compressive ductility testing of IN 738 LC superalloy revealed the behavior of the material under the two opposite loading conditions. According to Gleeble hot tensile ductility testing, a material loses all its ductility when the temperature reaches the on-heating nil-ductility temperature (NDT). The NDT is usually associated with materials loaded in tension. Practically, the material exhibits zero ductility at all temperatures above the NDT. The temperature range between the NDT and the liquidus T_L represents the on-heating zero ductility temperature range (ZDTR), within which the material exhibits zero ductility when loaded in tension. The T_L of IN 738 LC superalloy is around 1332 °C [15]. The result of Gleeble hot tensile ductility testing performed on IN 738 LC superalloy and presented in Table 1 showed that the NDT and the ZDTR for the alloy are 1160 °C and 172 °C, respectively. The material exhibited zero ductility at all temperatures from 1160 °C and above. In contrast to the hot ductility behavior of IN 738 LC superalloy when loaded in tension, all Gleeble specimens tested under compression at temperatures within the on-heating ZDTR under tension exhibited appreciable amounts of plastic deformation. For example, a Gleeble-tested IN 738 LC specimen that was compressed at the peak temperature of 1170 °C exhibited significant plastic deformation, where 48% increase in diameter was produced. Significant plastic deformation was observed in all compressed Gleeble specimens at temperatures above 1160 °C, up to 1230 °C. This ductility behavior of IN 738 LC superalloy under compression is in stark contrast to the behavior of the alloy under tension, where the material exhibits zero ductility at all temperatures from 1160 °C and upward. The result of this work suggests that, during hot ductility testing of IN 738 LC superalloy, the NDT that occurs at 1160 °C in materials tested under tensile loading is not applicable to materials tested under compressive loading. The material exhibits significant plastic deformation under compressive loading at temperatures above the NDT.

As discussed earlier, friction joining and forming operations in metal processing involve non-equilibrium heating, which induces drastic microstructural changes in the material. The effect of non-equilibrium heating on the dissolution behavior of the main strengthening γ' phase of IN 738 LC superalloy and the relationship between this dissolution behavior and hot ductility of the alloy were studied. These, and also including the phenomena associated with the behavior of the material under compressive and tensile loading, are discussed next.

4. Discussion

4.1. Non-equilibrium dissolution of γ' particles during rapid thermal cycle

The dissolution of $\text{Ni}_3(\text{Al,Ti})$, γ' , particles generally requires the dissociation of the solute atoms from the precipitates at the matrix/precipitate interface, followed by their diffusion away from the

interface into the matrix. At an extremely slow heating rate, corresponding to an equilibrium condition, it is expected that as the temperature increases the solubility of γ' forming elements in the matrix will increase gradually. In this situation, γ' particles will dissolve with increasing solute solubility of their constituent elements in the matrix such that γ' particles will dissolve completely, by solid-state dissolution, at the equilibrium solvus temperature of the particles. However, the rapid thermal cycle usually experienced during friction joining and forming processes departs significantly from equilibrium. For example, previous studies have suggested that heating rates during friction processing can be of the order of 200–300 °C s⁻¹ [16]. Under rapid heating conditions, the overall rate of dissolution of γ' particles will occur at a finite rate, which can be controlled by either or both of the two steps involved in the process viz., dissociation of precipitates into constituent elements and/or diffusion [17]. Several studies have shown that, under non-equilibrium rapid heating conditions, the temperature for complete dissolution of γ' particles increases with increasing heating rate, and the extent of departure from equilibrium dissolution behavior depends on the initial particle size [16,18,19]. Increase in complete dissolution temperature was found to be more pronounced with increase in particle size. An increase of about 120 °C in the complete γ' dissolution temperature at a heating rate of 8 °C s⁻¹ was reported for γ' precipitates with an initial size of 0.8 μm. A similar effect of heating rate on γ' solvus temperature has also been reported by DTA studies [20]. Therefore, depending upon the initial particle size and heating rate, the limited integrated time available for homogenization by diffusion process during continuous heating can cause γ' precipitate particles in nickel-based superalloys to persist to temperatures well above their equilibrium solvus temperature. SEM microstructural analysis of Gleeble-simulated materials, examples of which are shown in Fig. 2, revealed that, at a fast heating rate of 150 °C s⁻¹ and a holding time of 0.5 s, γ' precipitates survived up to 1250 °C, which is well above their equilibrium solvus of around 1160 °C.

The survival of γ' precipitates to temperatures above their equilibrium solvus under rapid heating can lead to non-equilibrium phase reactions below the solidus temperature. Notable among these is a non-equilibrium eutectic-type reaction between second phase precipitates and the matrix phase to produce a metastable liquid through constitutional liquation process, as first proposed by Pepe and Savage [17]. There exists a range of temperature in γ' precipitation-hardened nickel-based alloys within which γ – γ' eutectic reaction occurs, and persistence of γ' particles to this temperature range during continuous heating could result in their constitutional liquation [21]. In IN 738, γ – γ' eutectic transformation has been reported to occur over a range of temperatures, which could be below 1180 °C [22]. In the present work, the results of Gleeble simulation showed that γ' particles survived well above their solvus temperature to peak temperatures of up to 1250 °C during rapid thermal cycle. This consequently resulted in their constitutional liquation. It is known that a differentially etching thin film connecting γ' particles is an evidence of constitutional liquation [21]. An SEM micrograph showing constitutionally liquated γ' particles in a Gleeble-simulated IN 738 LC specimen, rapidly heated to a peak temperature of 1170 °C, is presented in Fig. 3(a). Furthermore, it was observed that constitutional liquation of γ' particles contributed, significantly, to intergranular liquation in the alloy. Fig. 3(b) shows the contribution of constitutionally liquated γ' particles to intergranular liquation ahead of a liquid film migration (LFM) zone in the alloy, with resolidified eutectic products formed along the liquated grain boundary. The formation of an LFM region, which is characterized by a homogeneously alloyed solidified zone produced by the migration process of liquid films similar to the one presented in Fig. 3 (b), has been previously observed at liquated grain boundaries of nickel-base superalloys and other alloys [23,24]. LFM was identified as an effective mode through which non-equilibrium grain boundary liquid can rapidly solidify at high temperatures. The most widely

Table 1
Hot tensile ductility data for IN 738 LC superalloy.

Parameter	Liquidus, T_L (°C)	NDT (°C)	ZDTR (°C)
Value	1332	1160	172

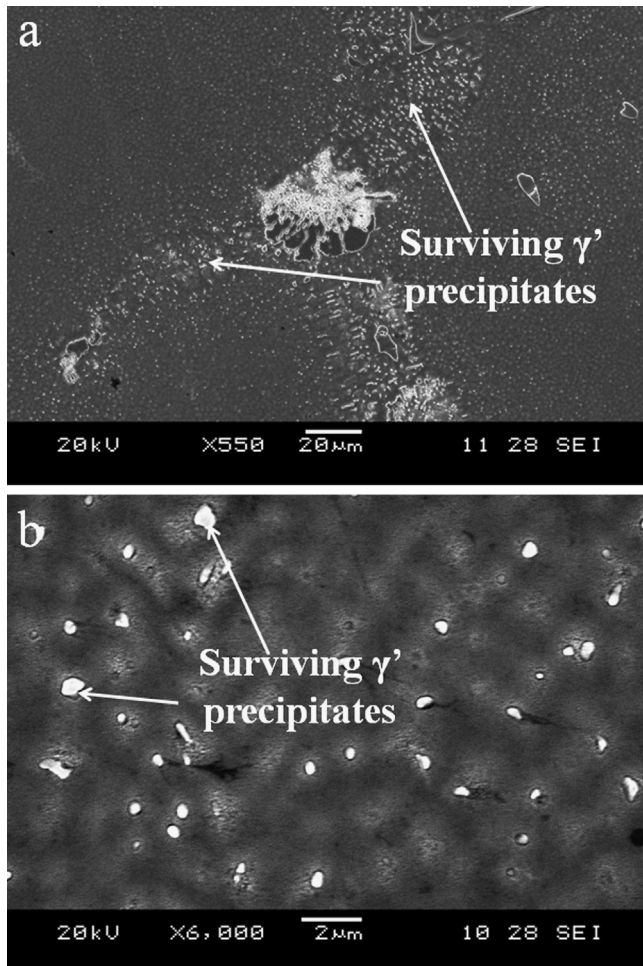


Fig. 2. SEM micrographs showing the survival of γ' precipitates to 1250 °C in a Gleeble-simulated IN 738 LC material.

accepted mechanism that is used in explaining LFM is diffusional coherency strain mechanism [25]. According to this mechanism, when a metastable liquid is introduced between two adjacent grains, a rapid equilibration process is set up by lattice solute back-diffusion. This causes a composition dependent variation in lattice parameter between the solute rich solid layer in contact with the liquid and the matrix well away from the solid–liquid interface. If the size difference between the solute and matrix atoms is sufficient, coherency strains could develop within the grains due to the lattice mismatch. The development of coherency strains on either side of the liquid film would cause shifts in the free energy curve of the liquid–solid interface such that the compositions of the liquid and solid at equilibrium will be different at both solid–liquid interfaces. This compositional gradient eventually results in LFM. Observations in this present work revealed that constitutional liquation of γ' particles and consequent intergranular liquation occurrence are possible during rapid heating of high temperature frictional processing and forming of precipitation strengthened nickel-base superalloys.

4.2. The behavior of a liquated material under tensile and compressive stresses

The observations made in this work show that IN 738 LC superalloy exhibited zero ductility under tensile loading above 1160 °C, where γ' precipitates dissolved by liquation reaction. The occurrence of liquation degraded the ductility of the material under tension and, as such, the material failed without plastic deformation. However,

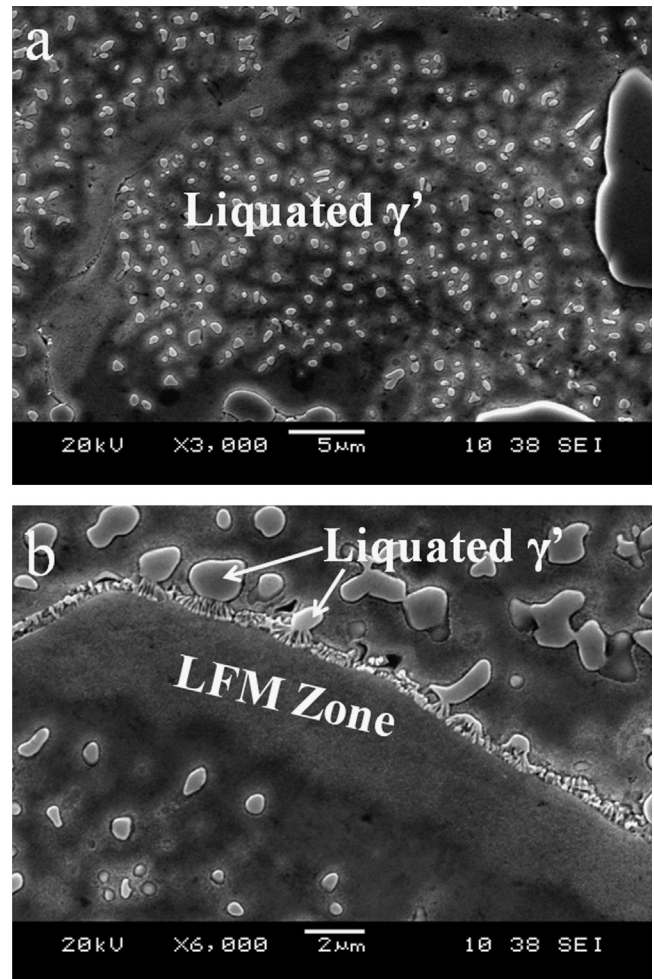


Fig. 3. SEM micrographs of a Gleeble-simulated IN 738 LC materials rapidly heated to 1170 °C and held for 0.5 s, showing (a) constitutional liquation of γ' precipitates, (b) liquid film migration (LFM) and the contribution of γ' liquation to intergranular liquation.

in contrast to the zero ductility behavior of the material under tensile loading, the material exhibited significant plastic deformation in the same temperature regime under compressive loading. The strain that was produced during plastic deformation of the material under compressive loading was observed to produce two important effects in the material. Firstly, strain-induced rapid re-solidification of the liquid that was produced by non-equilibrium heating was observed. Secondly, dynamically recrystallized grains, formed by considerable plastic deformation produced by compressive loading of the material, were observed. These observations are discussed next.

Compressive loading of the material during simulation induced compressive strains in the material. It has been suggested that such an imposed strain on solids can thermodynamically drive a non-equilibrium system towards equilibrium [26]. As discussed earlier, rapid temperature changes resulted in non-equilibrium phase reactions in the material. The kinetics of thermodynamically driven phase transformations in materials fundamentally involve atomic diffusion, which does not depend on temperature changes alone, but can be affected by both the magnitude and sign of externally imposed strain. Unfortunately, the effect of externally imposed strain on atomic diffusion in materials undergoing high temperature working operations is not usually accounted for. A rigorous analytical study has been used to establish a relationship between the activation energy per unit strain, Q' , and diffusion coefficients under strain, D (strain), and

without strain, D (relax), given by [27]

$$D(\text{strain}) = D(\text{relax}) \exp\left(\frac{-Q's}{kT}\right) \quad (1)$$

where, s is the strain (negative for compression and positive for tension), k is a constant and T is the absolute temperature. This relationship suggests that atomic diffusion can be enhanced by compressive strain and reduced by tensile strain. For example, the dissolution of γ' phase, which is typically known to be controlled by the diffusion of γ' forming elements away from the precipitate/matrix interface, was observed to increase under compressive loading in a nickel-base superalloy [28]. In addition, diffusion was experimentally confirmed to have increased under compression but decreased under tension in a Si–Ge material [29]. The same behavior was observed in a Cu–Si couple, where compressive loading resulted in significant increase in diffusion [30]. Studies have shown that the metastable liquid produced by non-equilibrium heating can isothermally resolidify through solid-state back-diffusion of solutes away from the liquid phase [31]. The effect of imposed compressive strain on back-diffusion-controlled isothermal resolidification of metastable liquid was investigated by the present authors in a previous study [32]. In that study, non-equilibrium eutectic-type liquation reaction was produced between γ' precipitates and γ matrix by rapidly heating IN 738 LC superalloy to temperatures above the equilibrium γ' solvus in the alloy ($\approx 1160^\circ\text{C}$), up to 1230°C . Applying different levels of compressive strain at various peak temperatures and holding times resulted in significant strain-induced rapid re-solidification of the metastable liquid. Fig. 4(a) shows the microstructure of a Gleeble-simulated material that was rapidly heated to 1230°C and held for 10.5 s without the application of compressive stress. In comparison, Fig. 4(b) shows the microstructure of another Gleeble specimen rapidly heated to the same temperature of 1230°C , held for 0.5 s and then compressively stressed to 30% length reduction in 10 s. The result shows that the re-solidified eutectic products that formed as a result of liquation reaction (Fig. 4a) were eliminated in the material on the application of a compressive load (Fig. 4b). This behavior has also been observed in another precipitation-strengthened nickel-base superalloy [33]. The occurrence of strain-induced rapid solidification in IN 738 LC superalloy suggests that the metastable liquid produced by non-equilibrium heating of the material to the processing temperature during friction joining and forming is rapidly re-solidified on the application of compressive load. In this case, a substantial part of the liquated material would be converted back into solid state and remain essentially solid at the processing temperature, thereby enhancing the materials workability. This behavior is contrary to the case of tensile loading, where decohesion occurs at the weaker liquated regions in the material, resulting in failure.

Another finding in this work is the observation of dynamic recrystallization in the materials tested under compressive loading

at temperatures above 1160°C . Fig. 5 suggests the formation of recrystallized grains in an IN 738 LC material rapidly heated to 1200°C , where 20% length reduction was imposed within 10 s. In order to confirm this occurrence, electron back-scattered diffraction (EBSD) based grain orientation analysis was carried out. Fig. 6 is a map of the recrystallized material, which shows new grains with an average size of about $5\text{ }\mu\text{m}$ in random colors. The average size of the newly formed grains is significantly less than the grain size of as-cast and solution heat treated IN 738 LC superalloy, which is in the order of hundreds of micrometers, up to about $2000\text{ }\mu\text{m}$. Dynamic recrystallization was reported to occur in another precipitation strengthened nickel-base superalloy at 1075 , 1100 and 1125°C and at strain rates as low as 0.01 s^{-1} [6], which is in the same order of magnitude as the strain rates used in this present work. Likewise, hot working of several precipitation strengthened nickel-base superalloys has been shown to be accompanied by significant dynamic recrystallization, which had considerable influence on hot workability of the alloys [34–36]. Dynamic recrystallization is generally known to be a completely solid-state phenomenon. As discussed earlier, significant intergranular liquation occurred in IN 738 LC superalloy at temperatures above 1160°C . This suggests that the new grains formed only after strain-induced rapid re-solidification at intergranular regions that have undergone liquation during rapid heating. The observation of dynamic recrystallization in IN 738 LC superalloy during hot compressive ductility testing above 1160°C is in agreement with significant plastic deformation experienced by the material within

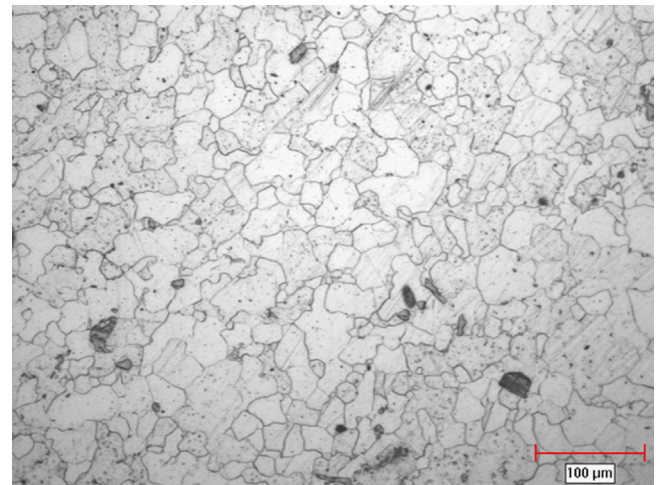


Fig. 5. Light-optical image of a Gleeble-simulated IN 738 LC material that was rapidly heated to 1200°C and compressively strained to 20% length reduction in 10 s, showing dynamic recrystallization.

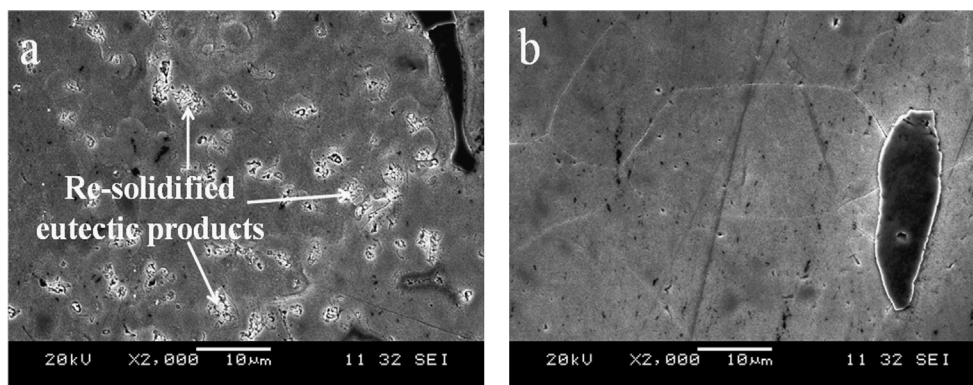


Fig. 4. SEM micrographs of a Gleeble-simulated material that was rapidly heated to 1230°C and (a) held for 10.5 s, followed by air cooling (b) held for 0.5 s and compressively strained to 30% length reduction in 10 s, followed by air cooling.

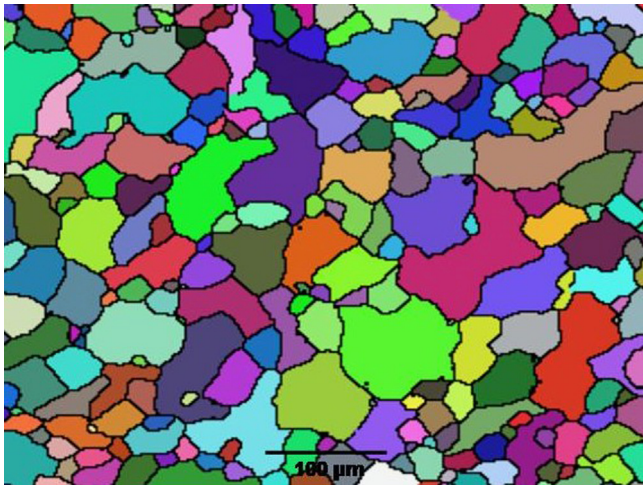


Fig. 6. EBSD grain map showing recrystallized grains in the Gleeble-simulated IN 738 LC material that was rapidly heated to 1200 °C and compressively strained to 20% length reduction in 10 s.

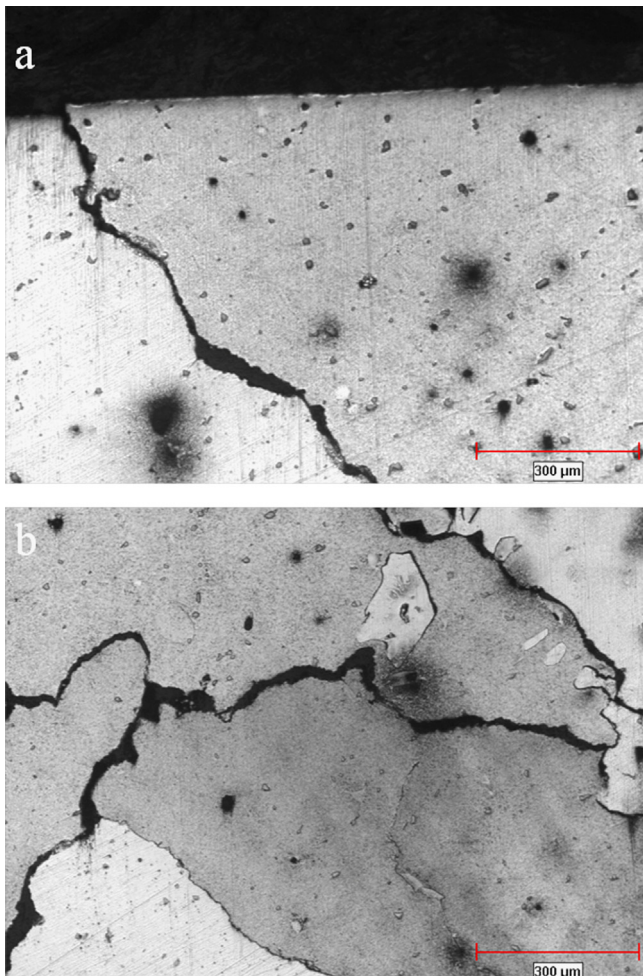


Fig. 7. Light-optical image of a Gleeble-simulated IN 738 LC material that was rapidly heated to 1170 °C, where tensile stress was applied. (a) An intergranular crack extending to the surface and (b) intergranular cracks in the interior of the specimen.

this high temperature regime. Fig. 7(a and b) shows optical micrographs of an IN 738 LC material that failed under tension at 1170 °C, showing intergranular cracks that resulted from liquation. Recrystallization was not observed in this Gleeble-simulated

material. Therefore, in contrast to the compressive ductility behavior of the material, lack of plastic deformation under tensile loading prevented dynamic recrystallization.

5. Summary and conclusions

1. Non-equilibrium heating, usually experienced during frictional processing and forming of precipitation strengthened nickel-base superalloys, induced drastic microstructural changes including constitutional liquation of γ' particles in IN 738 LC superalloy.
2. Constitutional liquation of the strengthening γ' phase and the contribution of this liquation reaction to intergranular liquation in the alloy caused degradation in hot ductility of IN 738 LC superalloy under tensile loading.
3. Contrary to liquation-induced zero ductility exhibited by the material under tensile loading at temperatures above 1160 °C, the alloy exhibited significant plastic deformation within the same temperature regime under compressive loading.
4. Considerable plastic deformation exhibited by the material under compressive loading above 1160 °C resulted in strain-induced rapid solidification of metastable intergranular liquid and formation of dynamically recrystallized grains, which are vital during high temperature processing of γ' precipitation strengthened nickel-base superalloys.

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