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Influence of heat treatment on microstructure and tensile behavior of a hot isostatically pressed nickel-based superalloy

Chunlei Qiu[1*], Xinhua Wu[1], Junfa Mei[1], Paul Andrews[2], Wayne Voice[2]

[1] School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

[2]Rolls-Royce plc, Derby, DE24 8BJ, UK

Abstract

A nickel-based superalloy powder RR1000 has been hot isotatically pressed (HIPed) and heat treated to produce different microstructures. Microstructures were investigated using a scanning electron microscope (SEM). Tensile testing was performed at room temperature and 700ºC and the deformed samples were examined using SEM and transmission electron microscope (TEM). It was found that in the as-HIPed condition the microstructure consisted of coarse and irregular-shaped primary and secondary γ' together with a low volume fraction of fine γ' (<50nm in diameter). Solution treatment below the γ' solvus followed by air cooling resulted in the formation of finer cuboidal secondary γ' (350nm-750nm) and medium-sized spherical tertiary γ' (100nm-200nm). This led to an improvement of both the 0.2% yield strength and ultimate tensile strength. Ageing of the solution-treated or of the as-HIPed samples at 760°C resulted in the precipitation of a high population of fine γ' (around 50nm) which further increased the strength. Within the resolution limit of the current TEM analysis, deformation at room temperature seemed to occur mainly by dislocations cutting through secondary γ' and very fine γ', accompanied by the formation of stacking faults within these precipitates; most of the medium-sized tertiary γ' precipitates in solution-treated and aged samples were not cut through but were surrounded by dislocations. Deformation at 700℃ happened by dislocations cutting through γ'

precipitates and γ matrix, leading to the formation of extended stacking faults across both γ and γ′. It is suggested that the optimum treatment of the powder superalloy is to solution-treat and age after HIPing; this results in properties which are comparable with those of thermomechanically processed samples.

Key words: Hot isostatic pressing; Heat treatment; Nickel-based superalloy; Microstructure; Deformation; Tensile fracture

*Corresponding author. Tel: +44 121 414 5163; fax: +44 121 414 7890; e-mail:
c.qiu@bham.ac.uk

1. Introduction

The need for jet engines to operate at higher temperatures to increase efficiency requires the development of nickel-based superalloys containing increased amount of the main strengthening elements such as Ti, Al, Mo, W, Ta and Hf. However, increased alloying usually leads to extensive segregation in the cast ingots which results in significant reduction in their hot workability. The decrease in workability adversely affects the overall component cost and the ability to achieve the desired properties. This has spurred the development of powder metallurgy (PM) techniques for producing components of highly alloyed compositions to achieve the desired microstructures, properties, and cost effectiveness. At present the nickel-based superalloy powders are HIPed to produce a billet after which it is processed following almost the same route as an ingot–involving extensive extrusion, forging and machining so that any prior particle boundaries (PPBs) or large inclusions are broken down. However, this approach results in significant cost increase because powder is more expensive than the ingot and the fly-to-buy ratio is equally bad for this powder-route as for the ingot-route.

Net-shape HIPing [1-5] offers a possible solution to this high cost of producing components from powder since this would lead to the reduction in forging and machining operations and thus to considerable cost reduction. The interest in this technology has been further spurred by the remarkable achievement in dimensional accuracy control in recent years through modeling [6-9]. However, directly HIPed nickel-based superalloy powder usually suffers from PPBs decorated with carbides, oxides or oxycarbides which are harmful to mechanical properties, especially the hot ductility, low cycle fatigue and stress rupture properties [10-14]. Therefore, it is very important to find out proper processing conditions for net-shape HIPing of nickel-based superalloy powder.

Previously, a HIPing condition which minimized the harmful PPBs has been developed for RR1000 alloy powder [15]. However, the mechanical properties (especially tensile strengths) of as-HIPed RR1000 samples could not meet actual requirements due to the development of irregular and coarse γ´ during slow cooling from HIPing temperature [15]. May et al [5] reported that with proper post-HIP heat treatments, desired mechanical properties could be achieved on as-HIPed RR1000. However, the influence of heat treatment on microstructure and mechanical behavior of HIPed RR1000 samples has not been fully understood and requires more investigation. In this paper, post-HIP heat treatments are performed and their influence on microstructure and tensile properties together with the deformation and fracture behavior of the HIPed and heat treated samples are studied.

2. Material and Methods

The powder used in this study is an argon-atomised nickel-based superalloy powder RR1000 which has a size range below 53μm in diameter. The chemical composition of this alloy is listed in Table 1. The γ´ solvus (S) of this alloy is around 1160°C.

Previous work [15] suggested that the optimum HIP temperature for this alloy is (S+20)°C. Thus, in the present study, samples were HIPed at (S+20)°C and at a pressure of 100MPa for 4 hours, followed by cooling at 5°C/min to room temperature. HIPing was carried out on stainless steel cans with a length of 180mm and an inner diameter of 45mm which after filling with powder, to a tap-density of between 60 and 65%, was outgassed and sealed by closing the evacuation tube by welding. Some HIPed samples were solution-treated for 2h at (S-40)°C and S, respectively, after which the samples were air cooled (the cooling rate was measured to be around 105º C/min). Solution treated samples were then aged at 760°C for 16h and air cooled. Some HIPed samples

were simply aged at 760°C for 16h and air cooled without a prior solution treatment. Details for different processing conditions are listed in Table 2. The samples were polished and chemically etched in activated colloidal silica solution and cleaned in acetone and then examined using a JEOL 7000 FEG-SEM microscope to study the structural integrity. Electron back scattered diffraction (EBSD) was also conducted in the SEM to study the grain structure. The samples were tilted 70° for EBSD mapping. The accelerating voltage was 20 kV and the working distance was around 18 mm. A step size of 10nm and a pattern resolution of 128x128 were used to develop the EBSD maps which were then analyzed using OXFORD INCA software supplied by OXFORD INSTRUMENTS to obtain the grain structure information such as the average grain sizes.

For observation of γ', the polished samples were further electrolytically etched in 10% H_3PO_4 in H_2O at 5V for 2-5s. The γ' precipitate sizes were measured by drawing a circle around the γ' particles using Image J software and then taking the diameter of the circle as an equivalent diameter for each precipitate. At least twenty particles from each colony of γ' have been measured and a range of precipitate sizes is given based on the measurement to define each distinctive class of γ'. The samples used for characterisation of γ' and grain structure were taken from cross sections of the HIPed bars. Given that powder particles and thus the grains are randomly distributed in the cans, the as-HIPed microstructure from these sections would be representative for the whole bar samples.

Tensile tests were performed at room temperature and 700°C using a computer-controlled electric screw driven Zwick/Z100 tensile testing machine on as-HIPed or HIPed and heat treated samples with a cylindrical geometry and a surface finish of 1.6μm. The tests were conducted at a

strain rate of $5x10^{-4}$ s^{-1}. In terms of testing at 700ºC, the samples were dwelt at 700°C for at least half an hour prior to testing. Tensile fracture surfaces were examined using SEM.

Discs for TEM examination were cut from the tensile-tested samples close to the fracture surfaces and normal to the tensile testing direction, followed by grinding to around 200µm thickness and then electro-polishing at -10°C and 25 V in 10% perchloric acid in methanol. TEM study on the dislocation structure of the tested samples was carried out under two-beam conditions or using the weak beam technique in an FEI TecnaiF20 FEG TEM microscope. An accelerating voltage of 200kV was used for TEM analysis.

3. Results

3.1 Influence of heat treatment on microstructure of HIPed and heat-treated samples

Fig. 1 show the microstructure of as-HIPed samples and of HIPed samples that were heat treated. It can be seen that heat treatments especially solution treatment show significant influence on microstructure. In as-HIPed sample, a microstructure consisting of large primary γ′ at grain boundaries, irregular-shaped intragranular secondary γ´ and very fine spherical tertiary γ′ (<50nm in diameter) could be observed (Fig.1 (a-b)). Ageing treatment generally shows no influence on primary and secondary γ′ in both as-HIPed and solution treated samples but seems to increase the population of fine tertiary γ′ (Fig.1 (d)). Solution treatment at (S-40)°C (STA) plus ageing considerably decreased the volume fraction of secondary γ′ and changed the irregular-shaped γ′ in the as-HIPed samples into cuboidal or near-triangular morphology. The size range of secondary γ′ was also decreased from 600~1200nm in diameter in the as-HIPed sample to 350~750nm after STA. Moreover, STA plus ageing led to the formation of a large

volume fraction of medium-sized and near-cuboidal tertiary γ′ (100~200nm in diameter, Fig.1 (f)). Among the secondary and tertiary γ′, even finer γ′ precipitates (<50nm in diameter) could be observed, leading to a multi-modal γ′ distribution. Solution treatment at S (STB) plus ageing was found to be able to completely eliminate the coarse primary and secondary γ′ and lead to the formation of an even finer secondary γ′ (~300nm in diameter) microstructure (Fig. 1(g)) but this led to the development of irregular-shaped morphology for γ′ (Fig. 1(h)).

Fig. 2 show the detailed structure of γ′ after deep etching. The irregular-shaped γ′ seem to show a cauliflower-like morphology while the cuboidal secondary γ′ appear to consist of numerous superfine particle-like zones (about 50nm in diameter for each zone, Fig.2 (b)). Given that the samples had been cut, ground and polished before etching, the structure revealed here may be not only from the surfaces but also from the interior (cross sections) of the secondary γ′ since some of the precipitates may have been sectioned through during metallographic specimen preparation. The presence of these superfine zones in the secondary γ′ seems to suggest that the formation of secondary γ′ may be due to the coalescing of fine precipitates during cooling from solution treatment temperature, which is highly possible for high γ′ volume fraction Ni-based superalloys [16]. These zones may correspond to the traces of those coalesced precipitates. The revelation of these nano-zones through deep etching also seems to suggest that there may be some kind of chemical difference between these zones and the material in between them. As the etchant was mainly designed to etch γ phase out, the removal of material in between the nano-zones in this etchant suggests that the material in between these zones may contain less γ′ forming elements. Further study is required to fully understand this issue. A small volume fraction of fan-type γ′ structure was also present in the as-HIPed sample or HIPed and heat treated samples. Like the cuboidal secondary γ′, they are also composed of considerable nano-zones (Fig.2 (d)).

In terms of the grain size, samples after STA seem to show a larger grain size (with average diameter of 15µm) than those that were simply HIPed and aged (with average grain size of 12µm in diameter), as evident in Fig. 3.

Fig. 4 show the influence of post-HIP solution treatment temperature on the structural integrity of the samples. It can be seen the porosity level increased with solution heat treatment temperature. Actually, solution treatment at lower temperatures such as (S-40)°C did not lead to formation of porosity. However, with solution heat treatment temperature increased from (S-40)°C to S, considerable thermally induced pores with diameter of 1-3 µm were formed (Fig. 4(b)). The current result is consistent with previous reports [17-21] where it suggested that the thermally induced porosity level generally increased with solution treatment temperature. Given that the thermally induced pores are mainly located at grain boundaries, they are believed to be formed due to boron-induced incipient melting at grain boundaries [18-19].

3.2 Influence of heat treatment on tensile properties

Fig. 5 shows the tensile properties of as-HIPed RR1000 samples and the HIPed and heat treated samples which were tested at both room temperature and 700°C. At room temperature direct ageing led to only a minor increase (about 50MPa) in both the 0.2% yield strength and ultimate tensile strength (UTS) as compared with the as-HIPed samples, which is obviously due to the increased precipitation of fine γ′ during ageing (Fig.1 (d)). However, sub-solvus solution heat treatment (STA) plus ageing led to a significant improvement (around 200MPa) in 0.2% yield strength and (around 150MPa) in UTS but also caused a reduction in elongation by around 5%. Increasing solution treatment temperature from (S-40)°C to S showed no obvious change to the tensile properties.

At 700°C, samples that were solution-treated (STA) and aged again showed much higher 0.2% yield strength and UTS over the as-HIPed samples. Direct ageing did not lead to obvious improvement in UTS. The increase in testing temperature (from room temperature to 700°C) led as expected to a decrease of 100MPa in both 0.2% yield strength and UTS but to limited change in elongation.

It is noted that the tensile properties of the current HIPed and solution-treated and aged samples are comparable to the thermomechanically processed counterparts with similar heat treatment conditions, as shown in Table 3.

3.3 Microstructural assessment of the deformed samples

Fig. 6 and Fig. 7 show the microstructure of the samples that were deformed after tensile testing at room temperature and 700°C, respectively. It can be seen that the secondary γ′, irrespective of morphology, are all uniformly elongated along the tensile loading direction. Occasionally, pronounced shearing of secondary γ′ could be observed in these samples. For irregular-shaped γ′, slip seemed to occur mainly by cutting through their branches. Slip passed through fan-type γ′ precipitates by shearing the finger-like γ′. The slip steps show offsets of tens of nanometers showing that many dislocations had sheared through the precipitates and that within the resolution of these images the dislocations have slipped on the same plane. To be mentioned, the shearing was generally constrained within individual grains and did not extend beyond grain boundaries. This kind of shearing became even more common at 700°C (see Fig. 7).

Fig.8 and Fig.9 show TEM bright-field images and weak-beam images of dislocation structure in the samples that were tested at room temperature. Quite a few dislocations could be observed inside the secondary γ′, irrespective of morphology. Weak-beam images further show that

stacking faults are present inside the precipitates, as shown in Fig.8 (b), (d) and (f). Some incompletely extended dislocations could be also observed. For the medium-sized tertiary γ′ with a diameter of 100-150nm (formed after STA and ageing), most were found to be free of dislocations, as shown in Fig.9. Instead, looping of dislocations around these precipitates and the formation of stacking faults in the γ channels could be observed. Given that there are considerable fine precipitates embedded in the channels between secondary or tertiary γ′ (Fig.1(f)), the formation of stacking faults in the channels suggests that the fine precipitates may have been cut through by dislocations.

The samples tested at 700°C, were found to show an even higher density of stacking fault ribbons (Fig.10). Extended stacking faults were widespread through the secondary γ′ precipitates. Most of the medium-sized γ′ were also cut through by dislocations leaving stacking faults - in contrast to the observations at room temperature where these were hardly cut by dislocations (Fig. 9). Many of the stacking faults even extended across the γ channel into the adjacent tertiary γ′ precipitates and formed complex arrays of faults. To be noted, the secondary γ′ shown in Fig. 10(b) seem to show considerable bumps which may correspond to those nano-zones as observed in Fig. 2 (b).

3.4 Examination of the fracture surfaces of tested samples

Fig. 11 and Fig. 12 show the typical fracture surfaces of the samples tested at room temperature and 700°C, respectively. At room temperature, all the samples irrespective of processing condition failed with a flat fracture surface that is normal to the tensile loading direction (Fig. 11 (a)). This kind of fracture is usually believed to be dominated by plane strain conditions and suggests poor fracture toughness [24]. Chevron-shaped marking with ridges and valleys,

radiating from a site near the sample surface could be observed. The crack initiation occurred generally in a cleavage mode from sample surface (Fig. 11(b)). Crack propagation proceeded in a transgranular fracture mode with a few facets and voids present on the fracture surfaces. The voids were formed either due to crack propagation along those remnant PPBs (Fig. 11(d)) or inclusion clusters (Fig.11(e)).

Samples that were tested at 700℃ generally failed with a mixed fracture feature, i.e., part flat fracture surface in the centre and part slant fracture surface near the edges (Fig.12(a)). The flat fracture area could be due to plane strain conditions while the slant fracture part is because of plane stress conditions [24]. This fracture feature usually suggests intermediate fracture toughness. Cracking was shown to initiate at the area near the sample surface in a cleavage mode and then propagate to form many chevron-shaped markings on the flat fracture surface (Fig. 12 (a-b)). The crack propagation occurred in a very faceted mode (Fig. 12(c)) but occasionally via inter-particle debonding fracture (Fig.12 (d)). The latter is mainly due to cracking along the remained PPBs. As compared with the room temperature fracture, fracture at 700℃ is more faceted and crystallographic which suggests that the deformation at elevated temperature is more planar, consistent with the above observation on deformation. In contrast to the flat part of the fracture surface, the slant part of fracture surface shows a much more ductile fracture pattern characterised by massive dimples, as evident in Fig. 12(e).

4. Discussion

The discussion will focus on two related aspects of the observations; firstly, the ability to develop very different microstructures from that of as-HIPed superalloy powder by post-HIP

heat treatments and secondly, the influence of the microstructures of the HIPed/heat treated powders on properties.

Subsolvus solution treatment (STA) plus ageing was found to change the microstructure of as-HIPed samples significantly. The dominant coarse and irregular-shaped primary and secondary γ′ precipitates in as-HIPed samples were replaced with a large volume fraction of finer cuboidal secondary γ′ and spherical tertiary γ′. Some of the coarse precipitates present in the as-HIPed samples in fact remain after solution treatment because this temperature is below the γ′ solvus. Solution treatment at higher temperature like S (STB) could even eliminate the large primary and secondary γ′ and replaced them with even finer and more homogeneous γ′ precipitates but led to development of irregular-shaped γ′ morphology. Moreover, according to previous work [17-21], if heat treatments are carried out at too high temperatures (like solution treatment at S), thermally-induced pores are formed which are not good for mechanical properties.

Both the 0.2% yield strength and UTS have been significantly improved after solution treatment and ageing, because of the high volume fraction of refined γ′ precipitates produced. The TEM study of the deformed samples generally showed behavior very similar to that reported in many papers of deformed Ni superalloys, with shearing through and looping of dislocations around γ′ precipitates. However some of the observations are inconsistent with previous reports. Thus in samples that were solution treated (STA) and aged and then mechanically tested at room temperature, most of the medium-sized tertiary γ′ (100~150nm) were found not to be cut through by dislocations whereas those much larger cuboidal secondary γ′ (350~750nm) were always sheared by dislocations. Increased precipitate size usually causes increased difficulty for dislocations to pass through the precipitates at room temperature and Orowan loops usually form around them [25]. Deep etching of the large cuboidal secondary γ′ revealed that these

precipitates actually consisted of many superfine zones with size of around 50nm which may correspond to the traces of those γ′ particles that may have come into coalescence during cooling from solution treatment (see Fig. 2(b)), which are smaller than the medium-sized tertiary γ′ precipitates (100~150nm). The materials in between these zones seem to contain less γ′ forming elements since they were etched out first during etching in the etchant which was designed to etch γ phase. The potential chemical variation within the secondary γ′ and the presence of the superfine zones may make it much easier for dislocations to cut through them since the distance that a ½<110> partial dislocation would have to traverse is limited to the size of these zones rather than the size of the secondary γ′. Further work may be needed to fully clarify this issue.

Another difference between the current results and previous reports lies in the nature of the dislocations in deformed samples. It is generally believed that at lower temperatures (<600ºC), deformation is dominated by coupled a/2<110> unit dislocations shearing the γ′ precipitates, with an APB in between them [26-28]. However, in the current study, coupled dislocations associated with APB were not seen in the γ′ precipitates in the samples that were tested at room temperature and instead stacking faults were observed. The most obvious explanation for this observation is that the alloy has a lower stacking fault energy than other polycrystalline nickel-based superalloys. This is possible since the superalloy investigated in the present study was designed to produce a minimum value of stacking fault energy through maximum addition of Co to promote planar slip and to inhibit cross slip, which are beneficial for both crack growth rates and creep behaviour [22].

At 700°C, the tensile deformation of HIPed and heat treated RR1000 was found to be mainly dominated by shearing of γ′ precipitates which led to the formation of stacking faults. Even those medium-sized tertiary γ′ which were rarely cut through by dislocations at room temperature,

were found to be easily cut at 700°C leading to well extended stacking faults. This appears to be inconsistent with previous reports on the deformation of polycrystalline nickel-based superalloys in the temperature range of 650ºC~750ºC where it is concluded that one of the deformation mechanisms such as microtwinning, isolated faulting, Orowan looping would be dominant depending on the applied stress level and microstructure [29-32]. However, it is also noted that microtwinning is a diffusion mediated process by which extended stacking faults are transformed by local diffusion into twins [27, 29]. It would be expected that at very high strain rates at the current study (as compared to creep deformation) there would not be sufficient time for local diffusion to occur and transform extended stacking faults into microtwins. As a result, extended stacking faults were observed in the current samples that were tensile tested at 700ºC.

Crack initiation of the current samples during tensile testing generally occurred in a cleavage mode and cracks propagated in a transgranular fracture mode at both room temperature and 700°C. Spherical pores or particles which may be due to fracture along PPBs were rarely observed, suggesting that the number of PPBs is very limited in the current samples. This is obviously beneficial for tensile properties especially hot ductility. Previous reports [13-15] suggested that the presence of considerable PPBs would easily lead to inter-particle debonding fracture during tensile testing and is particularly harmful for hot ductility. The current study demonstrates that with proper processing conditions, desirable microstructure and mechanical properties for net-shape HIPed Ni-based superalloys could be achieved.

5. Conclusions

- Refined microstructure and improved tensile properties of net-shape HIPed RR1000 could be achieved by conducting proper post-HIP heat treatments.

- Ageing treatment of HIPed RR1000 samples at 760°C led to increased population of fine γ′ and to the slight improvement in 0.2% yield strength at both room temperature and 700°C by around 50MPa.

- Solution treatment at 40°C below γ′ solvus together with ageing at 760°C completely transformed the coarse and irregular-shaped γ′ in as-HIPed RR1000 samples into finer cuboidal secondary γ′ and medium-sized tertiary γ′. This led to considerable improvement in both 0.2% yield strength and UTS. Solution treatment at γ′ solvus led to even finer but irregular-shaped γ′ precipitates and to the formation of thermally induced pores. This did not improve tensile properties.

- At room temperature, deformation of the HIPed and heat treated RR1000 occurred mainly via slip of dislocations through both secondary γ′ and very fine γ′ with the formation of stacking faults in these precipitates. Tensile deformation at 700°C was mainly due to the formation of extended stacking faults across both γ and γ′.

- Crack initiation of the HIPed and heat treated samples occurred in a cleavage mode while crack propagation proceeded mainly in a transgranular mode.

Acknowledgements

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