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Strain rate dependent microstructural evolution during hot deformation of a hot isostatically processed nickel base superalloy

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Isothermal constant true strain rate compression experiments coupled with electron back scattered diffraction (EBSD) analysis have been employed to understand the role of strain rate on the microstructural evolution during hot deformation of a hot isostatically processed nickel base superalloy. The flow behaviour of the alloy deformed at constant deformation temperature (1150 °C) and varying strain rates (0.001 s^{-1}, 0.01 s^{-1}, 0.1 s^{-1}, 1 s^{-1}) revealed two distinct strain rate dependent deformation characteristics. Microstructural observations revealed prevalence of superplastic flow behaviour preceded by dynamic recrystallization (DRX) with dynamic grain growth at the lowest strain rate studied. Sluggish degree of DRX is observed at intermediate strain rates characterized by low volume fraction of DRX grains and dominant presence of deformation substructure bounded with diffuse grain boundaries. Accelerated DRX is noticed at the highest strain rate owing to the higher accumulated stored energy driven large misorientation gradient and adiabatic temperature rise. Discontinuous DRX is found to be the prevalent DRX mechanism in the investigated conditions, which is substantiated by the evidence of necklace type microstructure, serrated grain boundaries, misorientation gradient analysis and relative fraction of grain boundaries. Towards the end, the dependence of strain rate on the evolution of primary twin boundaries (Σ3) and higher order twin boundaries (Σ9 & Σ27) are studied.

1. Introduction

Nickel based superalloys are considered as candidate materials for elevated temperature applications owing to their exceptional combination of static and dynamic mechanical properties coupled with resistance to degradation in extreme environments [1–3]. These alloys are widely used for producing critical rotating components in aircraft and power generation turbines and other critical applications related to nuclear and chemical industries [2]. The emphasis on ever-increasing demand of stronger alloys has led to the development of superalloys with complex chemical composition containing heavy elements, which in turn has posed difficulties in obtaining segregation free, homogenous structured superalloys by conventional ingot metallurgy route [3]. The aforesaid difficulties have been overcome by the development of superalloys by powder metallurgy (P/M) route involving powder production by inert gas/vacuum atomization, powder consolidation by hot isostatic pressing (HIP)/hot extrusion followed by isothermal forging to produce near net shaped components [1]. Keeping in view of the requirement, a relatively damage tolerant experimental P/M superalloy exhibiting exceptional creep and low fatigue crack growth rates suitable for turbine disk application is realized by HIP route. This alloy is strengthened by precipitation hardening of simple cubic $L1_2$ structured γ′ precipitates enriched with Al, Ti alloying elements, which is distributed multimodally in austenitic γ parent phase.

Understanding the thermomechanical behaviour of superalloys at elevated temperatures is vital in order to obtain propitious microstructure and mechanical properties to meet the application

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requirements. The microstructural evolution is strongly sensitive to the processing parameters like deformation temperature, strain rate and strain. Hence it is necessary to understand and optimize the processing parameters to tailor the microstructures [4,5]. The hot deformation behaviour of superalloys is characterized by complex microstructural evolution due to the synergistic effects of work hardening (WH), dynamic recovery (DRV), dynamic recrystallization (DRX) and superplasticity [4–9]. The presence of different alloying elements in nickel based superalloys tends to lower the stacking fault energy owing to the formation of widely spaced partial dislocation. However, the lowering of stacking fault energy is mainly determined by the nature of the alloying elements [2]. In the presently investigated alloy the presence of higher Mo, Ti Cr and Co content is likely to reduce the stacking fault energy. Being low stacking energy materials, the nickel base superalloys generally exhibit sluggish recovery due to hindered cross slip and climb [10,11], however the occurrence of dynamic recrystallization decreases the deformation resistance yielding beneficial fine grained structures resulting in better hot-workability to produce components of required shape and size. Superalloys exhibiting superplastic flow has the added advantage of imposing large strain without cracking at relatively lower forging loads [12]. Softening by DRX during hot deformation is further classified into: i) discontinuous DRX (DDRX), ii) continuous DRX (CDRX), and iii) geometric DRX (GDRX) [13]. Although DDRX characterized by nucleation and growth of strain free grains is the most commonly reported softening mechanisms during hot deformation in superalloys, the occurrence of CDRX at certain deformation conditions have also been reported in various superalloys [14–17]. The prevalence of softening mechanism is mainly influenced by the imposed processing parameters. For instance DDRX was dominant at high deformation temperature and high strain rate, whereas CDRX was found to be prevailing at lower strain rates during hot working of ALLVAC 718 plus [16].

Among the processing parameters like temperature, strain rate and strain, the dependence of microstructural evolution on strain rate is complex, which needs greater attention. In IN 718 superalloy, the DRX process was reported to be accelerated at lower strain rates [18], meanwhile accelerated DRX was revealed at higher strain rates during hot working of IN625 alloy [15]. In a recent work [19] the DRX behaviour was found to be sluggish at intermediate strain rates, however accelerated at lower and higher strain rates in austenitic stainless steel subjected to hot deformation. From the above inferences, it is clear that systematical studies are required to establish the effect of strain rate on structural evolution. In our previous work [8], the constitutive models for describing the hot deformation behaviour of the considered P/M superalloy over range of temperatures (1000 °C–1200 °C) and strain rates (0.001 s^{-1} to 1s^{-1}) is established by appropriate constitutive models. The objective of the present work is to understand the effect of strain rate on the evolution of microstructural mechanisms of the experimental P/M superalloy during hot deformation. The deformed microstructures are analyzed by EBSD technique and the microstructural mechanisms evolved at different strain rates are analyzed by carrying out detailed analysis of grain boundary characteristics. The evolution of volume fraction of DRX and grain sizes are determined and correlated to the microstructural evolution mechanisms.

2. Experimental procedures

The chemical composition of the hot isostatically processed experimental nickel base superalloy is summarized in Table 1. Cylindrical hot compression specimens of ∅8 mm and height 12 mm are electro-discharge machined from the starting material received in as-HIPed condition. Small grooves are recessed out from the sample surfaces to retain lubricant during testing and edges are chamfered in order to prevent fold formation during hot compression testing. The samples are coated with a thin layer of Deltaglaze™ 151 coating to reduce frictional effects. Isothermal compression studies are carried out in a 100 kN capacity servo hydraulic high temperature high strain rate test facility. The test system is equipped with a control system wherein the actuator speed is controlled by an exponential decay equation varying with time to maintain constant true strain rate during compression testing. The test system equipped with a split type resistance-heating furnace having maximum operating temperature of 1450 °C is capable of carrying out constant true strain rate tests ranging between 0.0001 s^{-1} and 10 s^{-1}. The compression samples are heated to 1150 °C at a rate of 25 °C/min and held at that temperature for 20 min before hot compression. The samples are isothermally deformed to 50% reduction in height ($\varepsilon = 0.69$) at four strain rates ranging from 0.001 s^{-1} to 1s^{-1}. The deformed samples are quenched in water immediately after compression to preserve the deformation microstructure. In order to obtain the starting microstructure at deformation conditions just before hot compression, small samples are heat treated at 1150 °C for 20 min in a resistance heating muffle furnace followed by water quenching. The deformed samples are sliced along the compression axis for microstructural observation. The samples for SEM-EBSD analysis are mechanically polished carefully to obtain mirror like surface finish followed by electropolishing in a solution of 10% perchloric acid in ethanol at 20 V. Microstructural analysis is carried out using a fully automated Electron Backscatter Diffraction (EBSD) system (Oxford Instruments, UK) attached to a FEG-SEM (Carl-Zeiss, Model: Supra 40). For heat-treated samples (undeformed), EBSD scans are performed over an area covering 1100 μm × 1100 μm employing a step size of 2 μm. Meanwhile, for hot deformed samples, scans are carried out at a step size of 0.5 μm over an area of 450 μm × 450 μm. A minimum of 2–3 scans are performed at each condition and the acquired EBSD data is exported to TSL-OIM™ analysis software for post-processing analysis.

Table 1
Analyzed chemical composition of studied nickel based P/M superalloy.

Element	Cr	Co	Mo	Al	Ti	Nb	Hf	B	Zr	C	Ni
Wt %	11.4	13.5	5.6	4.7	4.4	<0.02	0.41	0.02	0.03	0.04	Rest

3. Results and discussion

3.1. Starting microstructure

The nickel superalloy investigated in the present study was received in hot isostatically pressed (HIPed) condition in the form of ~Ø 60 mm billet. The initial microstructure just before compression was obtained by heat treatment as mentioned in Section 2 and was characterized subsequently. The image quality band contrast map with high angle grain boundaries (HAGBs), low angle grain boundaries (LAGBs), and Σ3 twin boundaries highlighted by black, blue and red lines respectively are illustrated in Fig. 1a. The starting microstructure dominantly consists of equiaxed grains with few annealing twins and the average grain size estimated by linear intercept method is 8.94 ± 1.02 μm. The Σ3 twin fraction is determined to be 7.95%, which is relatively less than what is typically observed in other superalloys [9,14,17]. As evident, the microstructure also revealed the heterogeneous presence of marginally larger grains indicating the occurrence of grain coarsening when subjected to elevated temperature exposure. The grain