



# Effects of initial aging time on processing map and microstructures of a nickel-based superalloy

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## ABSTRACT

Hot compressive deformation behaviors of the aged nickel-based superalloy are studied under the deformation temperature range of 920–1040 °C and strain rate range of 0.001–1 s<sup>−1</sup>. Based on the experimental data, the processing maps are developed and correlated with the deformed microstructures of the studied nickel-based superalloy. The effects of initial aging time on the processing map and microstructures are discussed in detail. It is found that the processing map and microstructures are sensitive to the initial aging time. When the initial aging time is shorter than 12 h, the spherical and short needle-shaped  $\delta$  phases (Ni<sub>3</sub>Nb) can stimulate the occurrence of dynamic recrystallization and improve the hot workability, as well as decrease the final forging temperature of the studied nickel-based superalloy. However, when the initial aging time is increased to 24 h, the excessive long needle-shaped  $\delta$  phases appear and become the potential locations of wedge cracking, which easily leads to flow instability during hot deformation. The aged superalloy under 900 °C for 9 h or 12 h is suitable for the hammer forging process. The optimum deformation parameters for the hammer forging process are 1010–1040 °C and 0.1–1 s<sup>−1</sup>. The aged superalloy under 900 °C for 9 h can be used for the conventional die forging. Furthermore, the forging temperature should be controlled in the range of 980–1040 °C, and the strain rate should be lower than 0.1 s<sup>−1</sup>. The solution-treated superalloy or the aged superalloy under 900 °C for 6 h or 9 h is suitable for the isothermal die forging, and the optimum hot deformation parameters is 980–1040 °C and near 0.001 s<sup>−1</sup>.

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## 1. Introduction

Generally, the hot working processing parameters (deformation temperature, strain rate and strain) significantly affect the hot deformation behaviors of metals or alloys [1,2]. Meanwhile, several deformation mechanisms, such as the dynamic recrystallization (DRX) [3–5], metadynamic recrystallization (MDRX) [6–9] and static recrystallization (SDRX) [10–12], result in the complex microstructural evolutions [13,14]. Therefore, a good understanding of high-temperature flow behaviors, microstructural evolutions and processing maps is very important for optimizing the hot forming processes of metals or alloys [15,16].

Dynamic material modeling (DMM) aims to correlate the flow behaviors with microstructural evolution, flow instability and hot

workability. Based on the dynamic material model (DMM), the processing maps were developed by Prasad et al. [17]. In recent years, the processing map has been extensively accepted as a powerful tool for optimizing the hot working parameters and controlling the microstructures of metals and alloys [18–35]. Li et al. [18] established 3D processing maps for the extruded ZK60 magnesium alloy and determined two feasible hot working domains, i.e., the DRX and superplasticity domains. Lin et al. [19] established the processing maps to investigate the hot deformation behaviors and microstructural evolutions of 42CrMo steel, and the optimum hot working domain is identified as 1050–1150 °C and 0.01–3 s<sup>−1</sup>. Jenab and Taheri [20] established the processing maps of 7075 aluminum alloy, and determined the dynamic recrystallization domain as 400–450 °C and 0.001 s<sup>−1</sup>. Teng et al. [21] studied the hot deformation behaviors of 40Cr alloy by processing map and microstructural observations, and a domain of dynamic recrystallization was identified as 900–1050 °C and 0.01–10 s<sup>−1</sup>. By the processing maps, Lin et al. [22] studied the flow behaviors and microstructural evolutions of a typical Al–Zn–Mg–Cu alloy (7075 aluminum alloy), and found the optimum hot working

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domains are 623–723 K and  $0.001\text{--}0.05\text{ s}^{-1}$ . Momeni et al. [23] developed the processing maps for a duplex stainless steel, and the instability region was only observed in the temperature range of 1423–1473 K and the strain rate of  $100\text{ s}^{-1}$ . Abbasi and Momeni [24] analyzed the hot workability of Fe–29Ni–17Co, and developed the power dissipation and the instability maps of the studied material. Wen et al. [25] constructed the processing maps of a typical Ni-based superalloy aged under 900 °C for 9 h, and identified that the flow instability is related to the adiabatic shear bands and the evolution of  $\delta$  phase during hot deformation. Quan et al. [26,27] discussed the optimum hot working parameters for the as-extruded 42CrMo steel and 3Cr20Ni10W2 heat-resistant alloy, which were validated by DRX microstructures without any wedge crack. Xia et al. [28] studied the processing map of coarse-grained Mg–Gd–Y–Nd–Zr alloy, and the optimum hot working condition were determined. El Mehtedi et al. [29] analyzed the hot workability of GC15 steel by torsion tests under the temperature range of 1125–1000 °C and strain rate range of  $0.005\text{--}5\text{ s}^{-1}$ . Rajamuthamilselvan and Ramanathan [30], Liu et al. [31], Senthilkumar et al. [32] and Han et al. [33] developed the processing maps for 7075 Al/20% SiCp composite, as-cast 6Mo superaustenitic stainless steel, Al-based nanocomposite and 20Cr–25Ni superaustenitic stainless steel, respectively, and the optimized processing parameters were obtained. Samantaray et al. [34,35] studied intrinsic workability of the modified 9Cr–1Mo steel and a nitrogen enhanced 316L(N) stainless steel by processing maps, and the relationship between microstructure and hot workability were investigated through microstructural observations. Peng et al. [36] investigated the hot compressive deformation behaviors of TC4-DT alloy with an initial equiaxed  $\alpha+\beta$  structure, and two regions, one in  $\alpha+\beta$  phase field and the other in  $\beta$  phase field, were marked as unsafe when the strain rate is higher than  $1\text{ s}^{-1}$ . The optimum hot working parameters for Al6063/0.75Al<sub>2</sub>O<sub>3</sub>/0.75Y<sub>2</sub>O<sub>3</sub> nano-composite were identified, and the flow instability characteristics were validated by processing maps and micrographs [37]. Additionally, the processing maps were developed for some other important alloys or composites, such as Al–Cu–Li alloy [38], Al–Cu–Mg alloys microalloyed with Sn [39], DC cast Al–15% Si alloy [40], SiC particles reinforced metal matrix composites [41], as-cast AISI M2 high-speed steel containing Mischmetal [42], Aluminum–5 wt% B4C Composite [43], near- $\alpha$  titanium alloy IMI834 [44], extruded Mg–12Li–1Zn BCC alloy [45], as-cast Ti–43Al–4Nb–1.4W–0.6B alloy [46], (SiCp+Mg2B2O5w)/6061 Al hybrid and SiCp/6061 Al composites [47], Al–Cu–Li–Sc–Zr alloy [48], 7005 aluminum alloy [49], and 800H alloy [50].

Due to their excellent strength, good oxidation and corrosion resistance under elevated temperatures, nickel-based superalloys are widely applied in the critical parts of aviation and aerospace engines [51,52]. It is well known that the large and complex aeroengine parts, such as turbine disks and engine shafts, are manufactured by the multi-pass hot working processes. The final mechanical properties are sensitive to the hot working parameters, such as the deformation temperature, strain rate and strain [53]. However, due to the narrow forming temperature range, great deformation resistance and complex microstructures, the hot working parameters of nickel-based superalloy should be controlled in a precise domain. Therefore, investigating the effects of hot working parameters on the deformation behavior, hot workability and microstructural evolution are very important for the hot forming of superalloys [54–69]. Shore et al. [54] studied the hot workability of the cast and wrought Incoloy 901 produced by electro-slag remelting, and found that the better workability of wrought material is associated with the easier dynamic recrystallization, compared to the cast material. Lin et al. [55] studied the hot tensile deformation behaviors and fracture characteristics of a typical nickel-based superalloy (GH4169). Also, Lin et al. [56] discussed the effects of initial  $\delta$  phase on the hot tensile deformation behaviors and fracture characteristics in detail, and found that  $\delta$  phase can cause the

obvious work-hardening behaviors at the beginning of hot deformation, and then accelerates the dynamic softening by stimulating the dynamic recrystallization with further straining. Meanwhile,  $\delta$  phases and carbides are the nucleus for the formation of microvoids. Wang et al. [57] investigated the hot compressive behaviors and processing maps of X-750 Ni-based superalloy, and the optimum hot working processing parameters were identified. Etaati et al. [58,59] investigated the hot deformation behaviors of Ni–42.5Ti–3Cu and Ni–42.5Ti–7.5Cu alloys, and established the accurate constitutive equations to predict the flow stress behavior under elevated temperatures. Chen et al. [60] proposed a segmented model to describe the kinetics of DRX for a typical nickel-based superalloy, and confirmed that the dynamically recrystallized grain size can be well characterized by a power function of Zener–Hollomon parameter. Lin et al. [61,62] studied the hot deformation behaviors of a typical nickel-based superalloy (GH4169), and developed a two-stage physically-based constitutive model and a new phenomenological model to predict the flow stress, respectively. Ning et al. [63] studied the hot deformation behaviors of the post-cogging FGH4096 superalloy with fine equiaxed microstructure, and developed a phenomenological constitutive model to characterize the dependence of steady flow stress on the deformation temperature and strain rate. Also, based on the isothermal compressive experiments, Ning et al. [64] constructed the processing map for GH4169 superalloy with stick  $\delta$  phase. Cheng et al. [65] studied the hot deformation behaviors of hot-rolled IN718 superalloy, and their results showed that the flow curves exhibit weak softening under most deformation conditions, and intensive strain localization and dynamic recrystallization occurred in the deformed specimens. Wang et al. [66] characterized the high-temperature flow behaviors and microstructural evolution of delta-processed Inconel 718 superalloy, and found that the dissolution rate of  $\delta$  phase during hot deformation is much faster than that under the stress-free aging. Li et al. [67] investigated the effects of the hot working process and heat treatment on the creep property of GH4169 superalloy. Pickering et al. [68] found that the serrated grain boundaries in Allvac 718Plus result from the discontinuous precipitation of  $\eta$  phase (Ni<sub>6</sub>AlNb), and it is considered as the predominant precipitation mechanism. Li et al. [69] investigated the microstructural evolution and dynamic recrystallization nucleation mechanism of hot-deformed Inconel 625 superalloy. Although a number of studies have been conducted to investigate the hot workability and microstructural evolution of different superalloys, the effects of initial aging time on the hot workability and microstructural evolution are still not clear. Further analysis should be carried out to study the combined effects of the initial aging time, deformation temperature, strain rate and strain on the processing maps, as well as on the microstructural evolutions of the studied nickel-based superalloy.

In this study, the hot deformation behaviors of a typical nickel-based superalloy, which is mainly used in the turbine disk of aviation engines, are investigated by hot compression tests over wide ranges of deformation temperature and strain rate. Based on the dynamic material modeling (DDM), the processing maps of the aged superalloy are constructed. The optimum initial aging time and hot working parameters are identified for different forming processes, including the hammer forging, conventional die forging and isothermal die forging. In addition, the microstructural evolution is analyzed to validate the established processing maps.

## 2. Materials and experiments

A typical nickel-based superalloy used in this study with the chemical compositions (wt%) of 52.82Ni–18.96Cr–5.23Nb–3.01Mo–1.00Ti–0.59Al–0.01Co–0.03C–(bal.)Fe. The main strengthening effects in the studied nickel-based superalloy is attributed to

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