Materials Science & Engineering A 586 (2013) 350–357

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Influence of niobium solutes on the mechanical behavior of nickel during hot working

N. Matougui [a,b], D. Piot [a,*], M.L. Fares [b], F. Montheillet [a], S.L. Semiatin [c]

[a] École nationale supérieure des mines de Saint-Étienne (ENSMSE), Laboratoire Georges Friedel UMR CNRS 5307, Centre for Materials Science and Mechanical Engineering (SMS Centre), RMT Department, 158 cours Fauriel, F–42023 ST ETIENNE CEDEX 2, France
[b] Mechanics of Materials and Plant Maintenance Research Laboratory (LR3MI), Badji-Mokhtar-Annaba University, P.O. Box 12, 23000 Annaba, Algeria
[c] Air Force Research Laboratory, AFRL/RX, Wright-Patterson Air Force Base, OH 45433-7817, USA

ARTICLE INFO

Article history:
Received 27 May 2013
Accepted 30 July 2013
Available online 7 August 2013

Keywords:
Mechanical characterization
Nickel based superalloys
Thermomechanical processing
Dynamic recrystallization

ABSTRACT

An experimental program was performed to determine the rheology and influence of niobium additions to high-purity nickel on dynamic-recrystallization behavior during hot working. Various high-purity alloys were prepared (pure Ni and Ni–0.01, 0.1, 1, 2, 5 and 10 wt% Nb) and deformed to high strains by hot torsion to characterize the mechanical behavior within the temperature range from 800 to 1000 °C at (von Mises equivalent) strain rates of 0.03, 0.1 and 0.3 s^{-1}. A simple analytical method was proposed for predicting the strain-hardening and dynamic-recovery parameters in the classical Yoshie–Laasraoui–Jonas equation. By the means, the effect of niobium solutes on plastic flow was determined, thus enabling a reasonable fit for the flow curves for the entire range of solid solution Ni–Nb alloys.

1. Introduction

Nickel-base superalloys are frequently used for large forged parts in aerospace industry, such as turbine disks, blades, and shafts. A better understanding of their mechanical behavior and microstructure evolution during hot working is required for manufacturers to enhance the performance in service.

Dynamic recrystallization (DRX) plays a fundamental role in microstructure evolution during the hot-working of metals because it controls grain size and crystallographic texture to a large extent. Discontinuous dynamic recrystallization (DDRX) occurs during the hot deformation of a wide range of metals (*e.g.*, copper, copper alloys, austenitic carbon and stainless steels, nickel and nickel-base superalloys). However, the detailed mechanisms of DDRX have been much less investigated in nickel-base superalloys than in steels. This is partly because such alloys contain a large number of alloying elements which may interact in a complicated way during hot deformation, thus requiring description of phenomena such as solute drag [1,2] and Zener pinning [3], and their effect on grain-boundary migration [4].

An important characteristic which reflects the nature of the recrystallization mechanism is the shape of the flow curve measured under hot-working conditions. For DDRX, such curves typically exhibit a steady state (after some transient flow-stress evolution), which is generally considered to result from a balance between strain hardening, dynamic recovery, and grain-boundary migration [5]. In materials undergoing DDRX, the steady state is generally reached at moderate von Mises equivalent strains (typically $\bar{\varepsilon} < 1$), in contrast to materials exhibiting continuous dynamic recrystallization (CDRX), such as aluminum and ferritic steels ($\bar{\varepsilon} > 10$) [6,7]. An analysis of stress–strain curves enables the determination of key constitutive parameters such as the strain-rate sensitivity of the flow stress and the apparent activation energy. In the steady-state range, the flow stress, as well as the distributions of all microstructure parameters, becomes independent of strain.

Among the various parameters which are likely to modify the kinetics or even the nature of the DRX mechanism in nickel-base alloys, the chemical composition through its effect on solid solution and precipitation hardening is quite important. Therefore, a systematic investigation for a simple model system, *viz*., pure nickel with a range of niobium additions (within the solid-solution range), can provide important insight into the impact of solutes on the kinetics of DDRX of more complex commercial materials such as alloy 718. In this regard, the quantitative effect of niobium on hot deformation remains somewhat unclear. For example, it is well known that segregation resulting from ingot solidification with a spatial scale corresponding to that of dendrites ($\approx$200 to 400 μm) is not eliminated by commercial homogenization and hot-working practices. An investigation for wrought alloy 718, which contains 5 wt% Nb, for example, revealed spatial variations of approximately $\pm$0.2 wt% Nb, thus indicating that the bulk diffusion of

* Corresponding author. Tel.: +33 4 77 42 00 87; fax: +33 4 77 42 66 78.
E-mail addresses: NedjouaMAATOUGUI@yahoo.fr (N. Matougui), piot@emse.fr, David.PIOT@Mines-StEtienne.fr (D. Piot), FARES.Lamine@Univ-Annaba.org (M.L. Fares), Frank.MONTHEILLET@Mines-StEtienne.fr (F. Montheillet), Lee.SEMIATIN@WPAFB.AF.mil (S.L. Semiatin).

0921-5093/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2013.07.079

niobium is slow even during high-temperature deformation [8]. In other work, the estimated grain-boundary mobilities in two different lots of 718, containing 5 or 5.4 wt% Nb, were significantly different, suggesting that niobium solutes interact with grain boundaries, and thus can migrate with them or dislocations, or both [9].

The present work was undertaken to enhance the current understanding of the hot working of superalloys. The specific objective was to establish the influence of the level of niobium in solid solution on the hot-working behavior of high-purity nickel-base binary alloys. For this purpose, a series of wrought model materials comprising pure nickel and Ni–Nb alloys with a range of niobium contents was prepared and subjected to hot-torsion tests. By this means, the specific effect of niobium addition on plastic flow could be assessed in the absence of the possibly confounding influence of other alloying and impurities typically present in commercial alloys. The classical strain-rate sensitivity (m) and apparent activation energy (Q) parameters were derived from the stress–strain curves. Furthermore, the influence of niobium solutes on strain hardening and dynamic recovery was quantified in the context of a single-state-variable (Yoshie–Laasraoui–Jonas) constitutive equation.

2. Materials and experimental procedure

High-purity nickel was prepared from commercial, electrolytic-purity material with a chemical composition given in Table 1. Purification was performed by induction melting in a water-cooled, silver crucible under a high-purity argon-hydrogen atmosphere using a procedure developed previously [10]. The induction coil was moved ~10 times from the left to the right of the crucible and back to effect zone refinement. The entire process was repeated 7 times with intermediate cleaning of the device. As a result, the levels of C, S, N, and O impurities were reduced to ~5 weight parts *per* million (wppm) or less (Table 1). By contrast, the Fe content was not modified by purification. Nevertheless, the purity obtained was still much higher than that of the nickel used by Luton and Sellars [11].

Commercial purity niobium (99.9 wt%) was added to the melt for preparing a series of 7 Ni–Nb ingots, each weighing ~1.2 kg, using the same procedure. The nominal compositions (in wt%) were Ni, Ni–0.01 Nb, Ni–0.1 Nb, Ni–1 Nb, Ni–2 Nb, Ni–5 Nb and Ni–10 Nb. The final niobium content was determined by various methods including inductive-coupled plasma, atomic absorption, and spark-emission spectroscopy, while the oxygen and carbon contents were measured by the Leco® method. For example, the chemical analyses of the Ni–0.1 Nb and Ni–1 Nb model alloys are reported in Table 2. They appear reasonably homogeneous (relative spatial fluctuation of ~10%), but a small amount of Nb appears to have been lost during melt processing. Furthermore, based on the equilibrium phase diagram for Ni–Nb (Fig. 1, [12]) and the work of Duerden and Hume-Rothery [13], which revealed a Nb solubility limit of 12.5 at% at high temperature, it was concluded that the Nb for all of the program alloys was in solid solution in the hot-working temperature range of interest in the present work (800–1000 °C).

After solidification processing, each ingot was hot forged following furnace preheating at 1050 °C into cylindrical bar of 14-mm diameter and then further reduced by swaging at the same temperature to 10.7-mm diameter.

To characterize hot-working behavior, torsion testing was used because it is more suitable than compression or tension tests to achieve the large strains typical of steady-state flow under well-controlled strain-rate conditions. For this purpose, round specimens with a reduced (gage) section measuring 27-mm length and 6-mm diameter were machined from each of the swaged bars. All specimens were heated to the test temperature (800, 900, or 1000 °C), soaked for 15 min, deformed (at a surface equivalent strain rate of 0.03, 0.1, or 0.3 s^{-1}) to a surface equivalent strain of 5, at

Table 2
Nb, O, and C content of the Ni–0.1 Nb and Ni–1 Nb alloys determined by (1) inductive-coupled plasma, (2) atomic absorption, and (3) spark-emission spectroscopy. Measurements were taken at three locations in the ingot: a—1st part to be solidified during the last displacement of the induction coil, b—near the half-length, and c—last part to be solidified.

Alloy	Nb (wt%)				O ($mg\ kg^{-1}$)	C ($mg\ kg^{-1}$)
	ICP[1]	AA[2]	SES[3] 1	SES[3] 2	Leco	
Ni–0.1 Nb	0.092	–	–	a—0.070 b—0.082 c—0.091	6.8	3.9
Ni–1 Nb	0.953	0.79	a—0.66 b—0.76 c—0.79	a—0.742 b—0.797 c—0.826	4.5	6.2

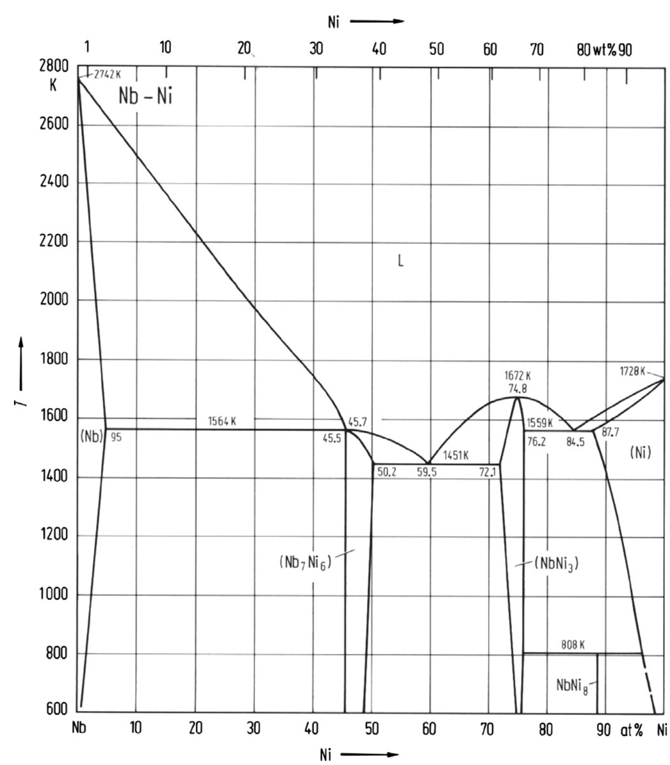

Fig. 1. Nb–Ni equilibrium phase diagram [12].

Table 1
Chemical composition of pure nickel used as alloying base (wppm, *i.e.* $mg\ kg^{-1}$, balance = Ni).

	As	Bi	Co	Cu	Fe	Mn	Pb	Sb	Si	Zn	C	S	O	N
As received (max. value)	0.5	0.2	0.5	10	150	–	0.5	0.5	5	0.5	150	15	–	–
Luton and Sellars [11]	–	–	–	100	500	100	–	–	100	–	40	–	–	–
After purification	–	–	–	–	150	–	–	–	–	–	≈5	≤5	≈6	≤5