Deformation Behavior and Microstructural Control of Co-Ni Based Superalloy

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ABSTRACT

In this doctoral dissertation, deformation and microstructure behavior in Co-33Ni-20Cr-10Mo superalloy are investigated at room and elevated temperatures. At elevated temperatures, hot forging is conducted by using hot compression tests at temperature range of 700-1200 °C and wide strain rates with constant strain of 0.5, while at room temperature, fatigue examination is carried out after 90% cold-rolling and heat treatment processes in Co-33Ni-20Cr-10Mo superalloy. The summary in every chapter study is explained as follows;

In Chapter 1 [Introduction], A superalloy, or high-performance alloy, is an alloy that exhibits excellent mechanical strength, creep and fatigue resistance at high temperatures, good surface stability, high corrosion resistance and oxidation resistance. There are several kinds of superalloy such as Ni-base superalloy and Co-base superalloy. The choice of cobalt-base superalloys in high temperature applications are primarily based on a good combination of superior tensile strength properties, excellent fabricability, weldability and good hot corrosion resistance for prolonged exposure. Co- base superalloys such as Co-Ni base superalloys also have been used in medical instrument parts, springs of small precision instruments and diaphragm. In addition, the physical metallurgy of Co-Ni base superalloys is very interesting but little understood. Therefore, many investigators attempted to clarify these problems. Also, it is interesting motivation to study deformation behavior and microstructure evolution in Co-Ni based superalloys at room and elevated temperatures due to limited information for these superalloys. Therefore, a Co-33Ni-20Cr-10Mo superalloy is investigated in this study with the purposes as follows; (1) To investigate deformation and microstructure behavior of Co-33Ni-20Cr-10Mo superalloy including construction of processing maps for optimal hot forging process; (2) To investigate deformation twinning due to Suzuki segregation in Co-33Ni-20Cr-10Mo superalloy at elevated temperatures; (3) To investigate effect of 90% cold rolling and heat treatment processes on the room temperature fatigue properties of Co-33Ni-20Cr-10Mo superalloy.

In Chapter 2 [Deformation and microstructure behavior of Co-33Ni-20Cr-10Mo superalloy during hot working], deformation and microstructure evolution are studied by carrying out compression tests between 950 °C and 1200 °C with increment of 50 °C at strain rates of 0.1, 1, 10 and 30 s^{-1} at constant strain 0.5. The flow curves obtained for the abovementioned strain rates in this temperature range show that this alloy has high work-hardening characteristics; this is due to the strong dislocation-solute interactions associated with dynamic strain aging (DSA). Microstructures deformed at temperatures up to 1050 °C consist of numerous deformation twins and highly dense dislocations, which are attributed to the high activation energy for deformation and the relatively low strain-rate sensitivity *m* in the temperature range of 950–1200 °C. Figure 1 shows deformation twinning and partial dynamic recrystallized grain nucleate at the twin boundaries at temperature 950°C and strain rate 10 s^{-1} with strain = 0.5. Dynamic recrystallization (DRX), which is dependent on the strain rate, occurs at T = 1000 °C, 1050 °C, 1100 °C, 1150 °C and 1200 °C.

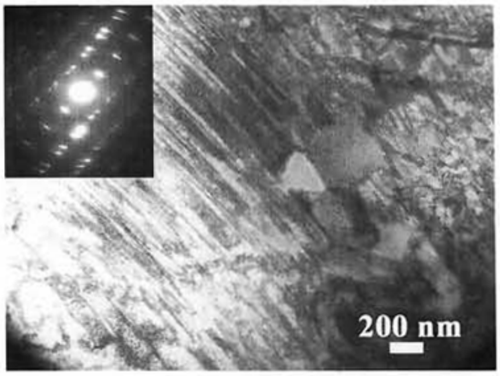

Fig. 1 TEM micrograph and diffraction pattern of Co-33Ni-20Cr-10Mo superalloy deformed by ε = 0.5 at T = 950 °C at $\dot{\varepsilon}$ = 10 s^{-1}.

In Chapter 3 [Constructing processing maps for hot working of Co-33Ni-20Cr-10Mo superalloy], hot forging process of Co-33Ni-20Cr-10Mo superalloy was carried out at temperatures ranging from 950–1200 °C and strain rates ranging from 0.01–30 s^{-1}. In order to obtain an optimum forging condition, various processing maps were constructed, such as a power efficiency map and an instability map, at different strain levels on the basis of a dynamic material model (DMM). Figure 2 and 3 show power efficiency map and instability map of Co-33Ni-20Cr-10Mo superalloy at 0.5 strain, respectively. At strain of 0.5, temperatures 1050–1200 °C and strain rates of 10–30 s^{-1}, DRX was observed with power efficiency values ranging from 35–44 %. Flow localization due to DSA and/or deformation twinning at temperatures 950–1000 °C and at low strain rates were observed to be in good agreement with those observed in the instability map.

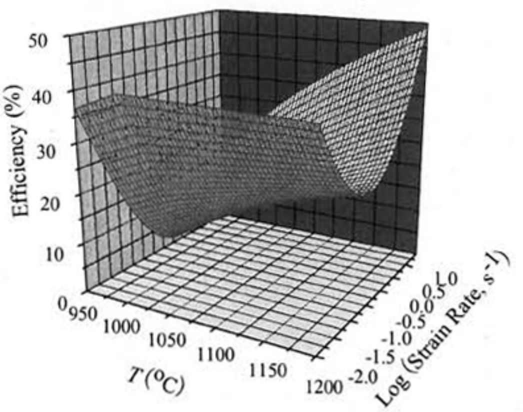

Fig. 2 Three-dimensional plot of efficiency power dissipation shows as a function of temperature and strain rate in Co-33Ni-20Cr-10Mo superalloy for a strain of 0.5.

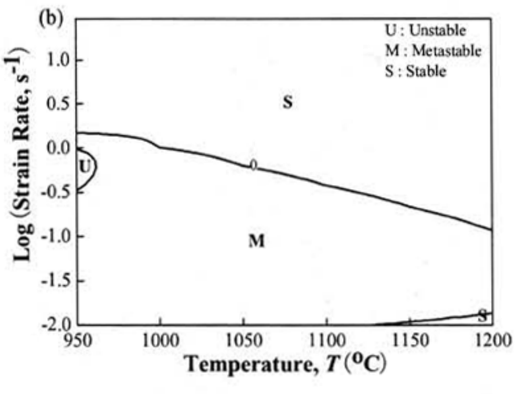

Fig. 3 Instability map of Co-33Ni-20Cr-10Mo superalloy show at ε : 0.5.

In Chapter 4 [Deformation twinning due to Suzuki segregation in Co-33Ni-20Cr-10Mo superalloy at elevated temperatures], dynamic strain aging (DSA) associated with deformation twinning has been investigated in Co-33Ni-20Cr-10Mo superalloy at T = 700-900 °C and $\dot{\varepsilon}$ = 10^{-3}, 10^{-2}, 10^{-1}, 1, 10, and 30 s^{-1} and ε = 50%. The flow stress curves showed high work hardening during straining and formed serrations at T = 700-850 °C and ε = 50%. These serrations increased with decreasing temperature and strain rate. When serrations occurred, negative strain rate sensitivity was obtained at those temperatures. Figure 4 shows 0.2% yield stress versus logarithmic strain rate of Co-33Ni-20Cr-10Mo superalloy at temperature range of 700-900 °C and constant strain of 0.5. Obviously, this phenomenon occurred in the present superalloy because of dynamic strain aging (DSA). This suggests that Co and Ni are segregated into the Shockley partials. At T = 700 °C and 900 °C and $\dot{\varepsilon}$ = 10^{-3} and 1 s^{-1}, as deformation proceeded to ε = 5%, 20% and 30%, planar dislocations array associated with pronounced stacking fault, stacking faults fringes due to Shockley partials and deformation twins were observed, respectively. In order to proof that deformation twinning is identical with Shockley partial or Suzuki segregation in Co-33Ni-20Cr-10Mo superalloy, TEM examination in [110] beam direction using **g** vector of **002** and $\mathbf{\bar{1}11}$ are available to observe partial dislocations formed at T = 700 °C, $\dot{\varepsilon}$ = 10^{-3} and ε = 20%.

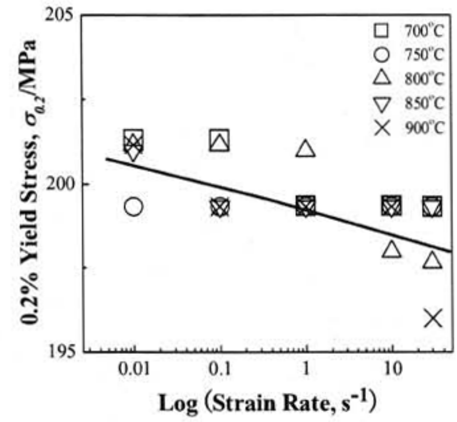

Fig. 4 Variation in $\sigma_{0.2}$ of Co-33Ni-20Cr-10Mo superalloy at T (°C) = 700–900 as a function of $\dot{\varepsilon}$.

In Chapter 5 [Effect of 90% cold rolling and heat treatment processes on the room temperature fatigue properties of Co-33Ni-20Cr-10Mo superalloy], room temperature fatigue properties of Co-33Ni-20Cr-10Mo superalloy after 90% cold rolling, aging and annealing were investigated in region between 10^4 and 10^7 cycles using R ratio of 0.1 with maximum stress from 0.75 to 0.3 UTS. The fatigue samples were analyzed in 0°, 54° and 90° to rolling direction under all conditions. According to the XRD examination, the direction of 0°, 54° and 90° to the normal rolling direction could be determined to be <001>, <111> and <110> directions, respectively. The anisotropic characteristic was obtained in high-cycle fatigue for samples with 90% cold-rolled and heat treatment processes. As can bee seen in Figure 6, TEM micrographs of 90% cold-rolled sample contain numerous fine deformation twins. These deformation twins are detrimental for Co-33Ni-20Cr-10Mo superalloy during cyclic loading because high stress concentration would be obtained between matrix and interface of deformation twins. Consequently, the brittle fracture mode occurred in 90% cold-rolled and aged. On the other hand, ductile fracture mode obtained in the annealed condition. Under this condition, deformation twinning can act as an initiation site of crack during high cycle fatigue due to low density of dislocation when compared to 90% cold-rolled and the aged condition. Furthermore, EBSD examination for 90° sample of aged condition showed that crack initiated and propagated along (111) plane, cryptographically revealed by analysis using Kikuchi patterns. This result is thought to be the reason why 54° sample of 90% cold-rolled and aged are showed lowest fatigue strength when compared to 0° and 90°. Figure 6 shows the *S-N* data curves that obtained anisotropy characteristic after 90% cold rolling and heat treatment processes in Co-33Ni-20Cr-10Mo superalloy.

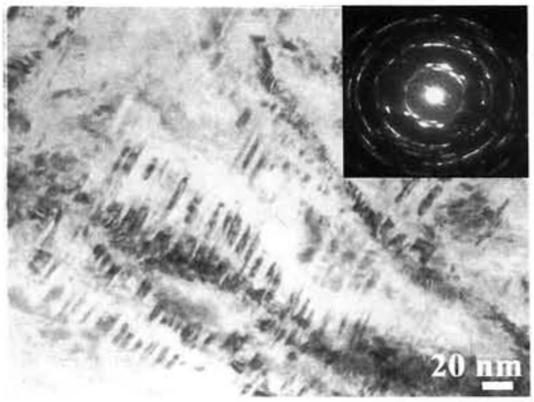

Fig. 5 Fine deformation twins show after 90% cold-rolling in Co-33Ni-20Cr-10Mo superalloy.

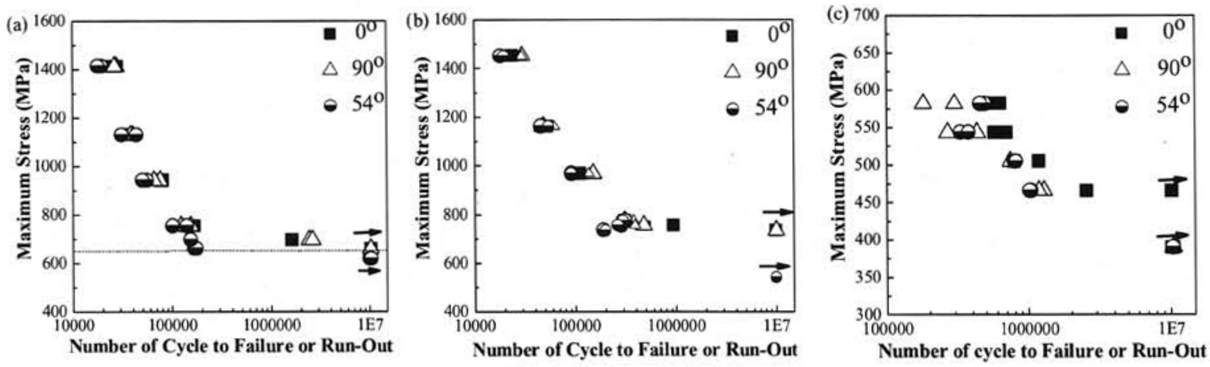

Figure 6 The *S* - *N* data curves of Co-33Ni-20Cr-10Mo superalloy with *R* ratio = 0.1 obtained as follow: (a) 90% cold-rolled, (b) 90% cold rolled and aged at *T*=500 °C-2h, and (c) 90% cold rolled and annealed at *T*= 1070 °C-2h.

In conclusion, this study systematically indicates the optimal processing method for microstructural control of Co-33Ni-20Cr-10Mo superalloy. In addition, the relation between Suzuki effect and occurrence of deformation twinning in the high temperature region where DSA takes place for the first time is investigated. Moreover, the fatigue strength is found to be improved by suppressing the initiation of the deformation twinning during loading. Thus, it is considered that this study is useful and worthwhile for actual application in industry as well as from the academic view point. Therefore, this study is considered suitable for doctoral dissertation.

論文審査結果の要旨

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