Serrated flow behaviors in a Ni-based superalloy

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Abstract

Serrated flow behaviors in Ni-20Cr-18W alloy were studied under different deformation conditions. It was found that there are no serrations observed on the tensile curves of the alloy at the temperature of lower than 250 °C and the strain rate of $10^{-3}$ s$^{-1}$. As the deformation temperature improves, the serrations first starts with the type A (250 °C) and transfers to the mixed type A + B (300 °C and 400 °C), then to the mixed type B + C (500 °C). Meanwhile, the serration behaviors are closely related to the strain rate and the strain temperature. It showed that when the deformation temperature is fixed, the serration curves starts with the type A ($10^{-3}$ s$^{-1}$), then transfers to the mixed type A + B ($10^{-2}$ s$^{-1}$) and eventually to type D ($10^{-1}$ s$^{-1}$) with the increasing strain rates from $10^{-3}$ s$^{-1}$ to $10^{-1}$ s$^{-1}$. Results showed that the serration behaviors of the alloy are related to the normal PLC effect. Combined with the McCormick’s model, the activation energy for the serrated flow behavior in this alloy is calculated to be 103.386 kJ mol$^{-1}$ and the value of $m + \beta$ to be 2.514, which suggests that the serrated flow of Ni-20Cr-18W superalloy is related to the interplay between the substitutional solute atoms and the movable dislocations.

1. Introduction

Due to excellent oxidation stability, outstanding corrosion resistance, good high-temperature creep resistance and strength, Ni-based superalloys have been frequently used in aircraft, aerospace, heat exchangers, chemical application and other fields [1]. In a certain temperature (especially in relatively elevated temperature) and strain-rate regime, some Ni-based superalloys can be deformed by sudden stress rise and down as serrations in the tensile curve, this phenomenon is also known as jerky flow or the PLC effect [2, 3]. Serrated flow behavior is a kind of heterogeneous plastic deformation phenomenon. It usually occurs in metallic glasses [4–6] and crystal materials especially in Al-based alloys [7–9], Ni-based alloys [10, 11] and steels [12]. Mulford and Kocks [13] investigated the strain rate sensitivity of the inhomogeneous plastic deformation for a Ni-based superalloy INCONEL 600. They modified the Slesewyk model and proposed the point that the dynamic strain aging (DSA) is responsible for the serrated flow, when solute atoms are waiting for thermal activation, they diffuse through dislocation [14–16]. Nalawade et al [17] investigated the serrated flow behavior in another Ni-based superalloy Alloy 718, results show that serrations appear owing to the diffusion of substitutional solute atoms Nb. Ni-based superalloys use nickel as the matrix, and the matrix phase is a stable austenite phase. The Ni-20Cr-18W superalloy is developed by enhancing the fraction of elements Cr and W. It can be strengthened by solid solution atoms and carbide precipitation. A large amount of W and Cr elements can not only act as solid solution atoms but also reduce stack faults of this alloy. The low stack faults can make matrix easier to produce twins which can also have important effects on the strength of Ni-based superalloy. Ni-20Cr-18W superalloy has good hot working deformation ability and welding performance, the working temperature can up to 1100 °C. Gao et al [18] studied the effect of grain refinement on mechanical properties of Ni-20Cr-18W superalloy, they found that small serrations appeared on the tensile curves from 450 °C to 600 °C. While both the deformation temperature and the strain rate can influence the serrated flow behaviors, resulting in different types of serrations on the
stress-strain curves. To have a deep understanding of plastic deformation mechanisms in Ni-20Cr-18W, the serrated flow behavior under different temperatures and strain rates has been studied in this paper, combined with McCormick’s model, the mechanism of the serrated flow is proposed.

2. Experimental procedure

The chemical composition of superalloy Ni-20Cr-18W used in this study is presented in table 1. The preparation process of this alloy is as follows: the alloy ingot is prepared by vacuum induction melting (VIM), the size is \( \varphi 110 \times 240 \text{ mm} \) after melting. Then use x-ray to inspect the ingot and remove the shrinkage hole. The heat treatment process with 1200 °C \( \times \) 24 h and furnace cooling is used to homogenize the ingot to reduce the segregation of chemical elements. The alloy ingot was forged within the range of 1100 °C to 1250 °C, the initial forging temperature is 1250 °C, the final forging temperature is not less than 1100 °C, recrystallization and stress relief annealing are required at 1250 °C. Subsequently, the ingot was hot-rolled between 950 °C and 1150 °C. Intermediate annealing was carried out at 1150 °C for 20 min, and finally the thickness of 3 mm rolled sheet was prepared. The samples used in subsequent tests were cut from the rolled plate by wire cutting. The samples are treated by solution treatment with a temperature of 1260 °C for 1h and then water quenching immediately. Tensile tests were performed at 25 °C (RT), 200, 250, 300, 400, and 500 °C with strain rate of \( 10^{-3} \text{ s}^{-1} \), tensile tests under different strain rates were carried out at 250 °C and 300 °C from \( 10^{-3} \text{ s}^{-1} \) to \( 10^{-2} \text{ s}^{-1} \) and to \( 10^{-1} \text{ s}^{-1} \). The electron backscatter diffraction (EBSD) was carried out to detect the textures. The morphology characteristics were performed via the scanning electron microscopy (SEM), and the elemental compositions of carbides were analyzed by the energy dispersive x-ray spectrometer (EDS).

3. Results and discussion

3.1. Initial microstructures

Figure 1(a) gives the SEM image of Ni-20Cr-18W alloy after solution treatment at 1260 °C for 1h. It can be seen that besides some residual carbides (as indicated by black arrows in figure 1(a), a small amount of annealing twins (as indicated by red arrows in figure 1(a) can be observed. The EDS analysis of carbides shown in the inset of figure 1(a) proves that the carbides rich in W, which indicates that the residual carbides located within the matrix are \( \text{M}_6\text{C} \) after solution treatment. Previous studies have reported that there are two types of carbides in the Ni-based superalloy, \( \text{M}_6\text{C} \) and \( \text{M}_{23}\text{C}_6 \). DSC analyses [19] show that the precipitation temperature of \( \text{M}_{23}\text{C}_6 \)
carbides ranges from 648 °C to 1147 °C, the carbides would be dissolved when the temperature is higher than 1160 °C. The precipitation temperature range of M6C carbides begins from 850 °C to 1210 °C, and would be completely dissolved into the matrix when the temperature exceeds 1390 °C, therefore when the alloy is heat-treated under 1260 °C for 1 h following by water quenching immediately, the M_{23}C_6 carbides would be dissolved
and M₆C carbides would be partly retained. The grain orientation distribution map is shown in figure 1(b). Combined with the standard pole figure, we can know that there is no texture detected after solution treatment, and the average grain size is about 3.2 μm by using a statistical measurement from Image Pro Plus software. Figure 2 shows its corresponding XRD pattern after solution treatment, indicating that this superalloy consists of the matrix γ phase and residual M₆C phase after solution treatment.

3.2. Serration behaviors associated with the deformation temperatures

Figure 3 shows the true stress-true strain curves of Ni-20Cr-18W under different deformation temperatures with a strain rate of 10⁻³ s⁻¹. It can be observed that when the temperature increases to more than 250 °C, the true stress-true strain curves of this alloy are serrated and characterized by different types depending on the deformation temperature. According to the previous work [20], different types of serrations have been defined, as depicted in figure 4. Type A serration is considered as the periodic serrations and can be distinguished by a sudden stress rise and then decline to below the normal level of the tensile curve. Type B serration is defined as the oscillations around the normal level of the tensile curve and flows in quick succession. It usually appears at the initial stage of stress yielding at high temperatures and low strain rates or evolves from type A serration with increasing strain. Type C serration often occurs below the normal level of the tensile curve and is characterized by the yield drops, which can be ascribed to the movable dislocation unlocking. Type D serration can be distinguished by the plateaus on the true stress-true strain curve, which can be attributed to the propagation of slip bands. Type E serration is usually following type A and is characterized by a wave on the curve. As shown in figure 3, the serration of type A is first triggered when the deformation temperature increases to 250 °C; a mixed serration of type A followed by type B can be observed as the deformation temperature increases to 300 °C and 400 °C, and when the deformation temperature improves to 500 °C, a mixed serration of type B followed by type C can be detected. The occurrence of type C is consistent with the work of Rodriguez and Venkadesan [21] in which they confirmed that type C is more like to be triggered than type A and type B under relatively high deformation temperatures.

Variations of ultimate tensile strength (σᵤ), yield strength (σₛ) and elongation to fracture, the magnitude of strain hardening (Δσ = σᵤ − σₛ) as a function of tensile temperature at a strain rate of 10⁻³ s⁻¹ are shown in figure 5. It can be seen that these curves of mechanical properties are not monotonous, the overall tendencies of ultimate tensile strength and yield strength are upward except for a sudden drop at 250 °C. In the temperature region where the serration appears, the elongation drops sharply at the onset and then increases slightly. As reported by Zheng et al [22], the brittleness of the alloy increases because the diffusing solute atoms at high temperatures could interplay with movable dislocations, more solute atoms could eventually reflect the dislocation movement. Thus with the increase of the aging temperature, more solute atoms would accelerate migration around the dislocations to hinder their further migration, eventually resulting in the early failure of the alloy. The overall tendencies of ultimate tensile strength and yield strength are rising, this phenomenon is contrary to the report of Gao et al [18]. It is well known that recovery at high temperatures can reduce the tensile strength of alloys, but it must be recognized that the effect of strain hardening during deformation cannot be ignored. The magnitude of tensile strength will depend on the relative degree of the recovery and strain hardening at high temperatures. In this experiment, only at 250 °C was the effect of strain hardening reduced,
indicating that strain hardening is dominant from 25 °C to 500 °C, this intense effect of strain hardening results in an increase in the tensile strength of this alloy. Meanwhile, the interplay between the dislocation entanglement leading to strain hardening and the solute atoms may result in the non-monotonic variation between elongation and strain rate, as shown in figure 6.

3.3. Serration behavior related with the strain rates

Figure 6(a) shows the true stress-true strain curve of Ni-20Cr-18W under various strain rates at 250 °C. It can be observed that at the strain rate of $10^{-1}$ s$^{-1}$, there are no serrations detected and the curve is smooth. When the strain rate decreases to $10^{-2}$ s$^{-1}$, a light oscillation can be observed which means the onset of type A serration, and typical type A serration is presented on the tensile curve as the strain rate decreases to $10^{-3}$ s$^{-1}$. Figure 6(b) gives the true stress-true strain curves under different strain rates at 300 °C. When strain rate is set to be $10^{-1}$ s$^{-1}$, it can be seen that the serrations of type D are induced and then evolve into type A serrations when the strain rate decreases to $10^{-2}$ s$^{-1}$. As the strain rate decreases to $10^{-3}$ s$^{-1}$, a mixed serration of type B followed by type A could be observed. One should note that there are two questions needed to be emphasized. First, the effect of strain rate on the types of serration under the same deformation temperature. Second, the effect of deformation temperature on the types of serration at the same strain rate, as deposited above in the section of 3.1. As for the former one, various strain rates will have a different influence on the generation and migration of mobile dislocation, and deformation twins could also be produced by tensile deformation. For the latter one, higher temperatures can change the diffusion rates of solution atoms and then influence the deformation behavior. Carbide precipitation is also a factor that cannot be ignored. All of these might have complicated effects on the types of serrated flow curves.

The detailed discussion could be seen in the following section.
3.4. The mechanism of serration flow behaviors

The movable dislocation moves faster than the diffusion of solute atoms when deformation occurs at elevated strain rates; the movable dislocation can’t be hindered effectively. This leads to the occurrence of type A serrations under the appropriate condition. With the decrease of strain rates, the pinning of solute atoms to the dislocations becomes significant and then type B serrations could be triggered. It should be noted that the oscillation amplitude of serration is higher at high temperatures and low strain rates. This can be attributed to the fact that the diffusion rate of solution atoms is faster at higher temperatures which makes more solution atoms to cluster around the dislocations and lower strain rates provide sufficient time for effective pinning of movable dislocation. These effects above lead to a higher amplitude of serrations at elevated deformation temperatures and declined strain rates.

The influence of strain rate on critical strain ($\varepsilon_c$) at different temperatures has been shown in figure 7. The critical strain ($\varepsilon_c$) increases with strain rate increasing and temperature decreasing, this relationship can be described as the normal behavior of the Portevin-Le Chatelier (PLC) effect [23, 24].

The critical strain ($\varepsilon_c$) with the normal behavior of the PLC effect has been represented by McCormick’s model [25]. In this model, the following three factors are considered: (i) increase of diffusion rate by vacancies produced during the plastic deformation; (ii) increase in the number of moving dislocations during the deformation; and (iii) aging of dislocations by solute atoms when they are waiting for the obstacles to pass. It is assumed that the aging time of solute atoms to the dislocations is equal to the waiting time of dislocations at the obstacles, the equation below can be acquired [26]:

$$\dot{\varepsilon} = B \varepsilon_c^{(m+\beta)} \left( \frac{C_s}{C_i} \right)^{\frac{1}{2}} \lambda \exp \left( -\frac{Q}{KT} \right)$$  \hspace{1cm} (1)

In this equation, $\dot{\varepsilon}$ denotes as the strain rate, $\varepsilon_c$ is the strain of first serration appears(critical strain), $m, \beta, \alpha$ ($\alpha = 3$) and $B$ are all constants, $C_s$ stands for the solute atoms concentration, and $C_i$ is the solute atoms concentration needed to pin the movable dislocation, $\lambda$ is the mean distance between obstacles, $Q$ stands for the vacancy activation energy, $K$ is the Boltzmann’s constant, $T$ is the deformation temperature.

Rodriguez proposed that the mechanisms of serrated flow can be identified by the exponent $m + \beta$[20]. The value $m + \beta$ is equal to the slope of the plot of strain rate versus critical strain in the log scale (figure 7). For substitutional solute alloys, typical $m + \beta$ values are between 2 and 3; for interstitial solute alloys, values are between 0.5 and 1. Since alloy Ni-20Cr-18W has residual carbides after solution treatment, it is appropriate to use McCormick’s model to evaluate the experimental results. Thus the calculated $m + \beta$ of the investigated alloy approximately to be 2.514, which indicates the substitutional solute atoms could be the main reason for observed serrated flow behaviors.

The plot of the relationship between critical strain and temperature of Ni-20Cr-18W alloy is shown in figure 8. It suggests that in the log scale the critical strain varies linearly with the temperature, and the value of critical strain is increased by decreasing the temperature at each strain rate.

![Figure 8. The plot of critical strain ($\varepsilon_c$) versus $1/T$ at different strain rates.](image-url)
energy (dislocations and precipitation phase) the serrated flow appears, which accelerates the diffusion of solute atoms and shortens the aging time. Finally, when two times are equal, the critical strain of moving dislocations is longer than the waiting time of dislocations. Therefore, the true stress-true strain curve exhibits serrated flow.

Regarding equation (1)

\[ \varepsilon^{m+\beta} = K \exp \left( \frac{Q}{RT} \right) \]  

(2)

In this equation, \( R \) is the gas constant, \( m + \beta \) can be determined at a certain strain rate, thus the activation energy \( (Q) \) could be calculated by the slope of critical strain in the log scale versus \( 1/T \) in figure 8. The result of activation energy for the serrated flow behavior in this alloy is calculated to be 103.386 kJ mol\(^{-1}\). Serrated flow behaviors can be observed in a large number of metallic materials, and its mechanisms can be attributed to several reasons below: (i) Strain-induced martensitic transformation; (ii) deformation twinning and (iii) the interplay between the solute atoms and movable dislocations [27]. Discontinuous deformation caused by Strain-induced martensitic phase transformation is proposed by Hiroyuki Kato and Kazuki Sasaki [28], they reported that in NiTi alloy, serrated flow curves caused by strain-induced phase transformation only appear in a narrow temperature range from 584 °C to 601 °C, outside this range all other curves are smooth. While in figure 3, the temperature range of serrated flow curves in Ni-20Cr-18W exceeds more than 200 °C, the amplitude of serration mentioned by Singh and Doherty [29] was as large as 80 MPa in MP35N alloy, they proposed that the composition of matrix changes due to solute atoms move from matrix fcc phase to cubic phase, this increases the difficulty of martensite nucleation and leads to larger and larger serrations amplitude until the matrix becomes fully stabilized against further plate formation. However, the largest amplitude of serration in this experiment is about 11 MPa, and the strain-induced martensite phase transformation has not been observed yet in this superalloy, so the serrated flow caused by Strain-induced martensite phase transformation in Ni-20Cr-18W is almost impossible. Serrated flow behaviors arose from deformation twinning has been reported before [10, 27, 30], but it should be noticed that most of the serrated flow caused by deformation twinning appears at low temperature or even at room temperature, while the serrated flow behavior in Ni-20Cr-18W did not appear below the temperature of 200 °C even though a small amount of annealing twins is observed. Gopinath et al [31] reported that the serrated flow behavior becomes prominence with the increase of temperature and the decrease of strain rate, this is the opposite of the condition in which twins are more likely to be produced, so it suggests that deformation twinning is not the reason of serrated flow behaviors in 720Li alloy. From the discussion above, it can be concluded that deformation twinning is not the reason to cause serration in superalloy Ni-20Cr-18W. The value 2.514 of \( m + \beta \) indicates the substitutional solute atoms might be the reason for the observed serrated flow behaviors in this alloy. According to McCormick’s theory [25], movable dislocations are impeded by various obstacles (solute atoms, forest dislocations and precipitation phase) during the plastic deformation. These movable dislocations are waiting to surpass the obstacles because of thermal-mechanical coupling. Because of the increase in thermal activation energy, the solute atoms begin to diffuse around the movable dislocations. If there are enough solute atoms around the dislocation that the dislocation can be pinned, the critical diffusion time of solute atoms can be called the aging time. At the onset of the tensile test, the diffusion rate is very slow, hence the aging time of solute atoms to movable dislocation is longer than the waiting time of dislocations. Therefore, the true stress-true strain curve is smooth. As the tensile test goes on, the dislocation density increases sharply due to the deformation, which leads to the waiting time increases. Meanwhile, the plastic deformation results in the increase of vacancy density, which accelerates the diffusion of solute atoms and shortens the aging time. Finally, when two times are equal, the serrated flow appears, figure 9 shows a schematic of the mechanisms of the serration flow behaviors. When at lower temperatures, the diffusion rate of solute atoms declines, resulting in an extended aging time. Therefore by increasing the strain, a longer waiting time and a higher vacancy concentration can be achieved. And at a higher strain rate, the movable solute atoms are unable to pin the dislocations effectively in a relatively short time. For the above reasons, the critical strain increases with decreasing temperature and increasing strain rate, as shown in figure 8. These discussions lead to the conclusion that the reasonable mechanism of serrated flow behavior in Ni-20Cr-18W is the interplay between the substitutional solute atoms and the movable dislocations.

Figure 9. Schematic diagram illustrating the generation of serrated flow.

\[ \varepsilon^{m+\beta} = K \exp \left( \frac{Q}{RT} \right) \]
In Ni-20Cr-18W superalloy, substitutional solute atoms are mainly Cr and W. Then which atom has the biggest effect on the interplay between solute atom and dislocation? The atomic radius of W is 0.141 nm, while the atomic radius of Cr is 0.127 nm which is very close to matrix Ni’s atomic radius of 0.124 nm. According to the report of Wang et al. [32], the diffusion rate of Cr is about 3.7 times faster than W in Ni-rich fcc Ni-Cr-W ternary system alloys. Rather than the effect of atom size mismatch, the diffusion rate of atoms at high temperatures seems to be the most important factor for dislocation pinning. Therefore, Cr has a greater influence on serrations than Gao et al. [18] attributed the serrations in high-temperature tensile curves of Ni-20Cr-18W to the interplay between carbide and dislocation, the volume fractions of carbides in their paper were 10% and 18% respectively. Cai et al. [33] also confirmed that serration amplitude increased with increasing carbides at a given temperature, while in this experiment, the volume fraction of residual carbides after solution treatment was about 0.26%, which indicated that the direct effect of residual carbides on movable dislocation is limited. It should be noted that the activation energy of serrated flow behaviors in this experiment is much smaller than the migration activation energies of Cr and W in Ni-Cr-W ternary system (Atomic mobility: $Q_{Cr} = 237.585$ kJ mol$^{-1}$, $Q_{W} = 344.562$ kJ mol$^{-1}$) [32]. From the discussions above, we know that during tensile deformation, the migration activation energy of solute atoms decreases due to the increase of vacancy density and dislocation. The precipitation phase can also affect the interplay among movable dislocations, forest dislocations, and substitutional solute atoms. these factors lead to a relatively low activation energy of this serrated flow behavior.

4. Conclusion

In this work, the serration behaviors of Ni-20Cr-18W superalloy were investigated under different conditions. The following conclusions can be drawn:

1. The serration behaviors of the alloy are influenced by the deformation temperature. At a certain strain rate of $10^{-3}$ s$^{-1}$, there are no serrations observed on the tensile curves of the alloy when the deformation temperature is lower than 250 $°$C. When the temperature is improved, the serration first starts with the type A(250 $°$C), then changes to the mixed type A + B(500 $°$C and 400 $°$C) and eventually to the mixed B + C(500 $°$C).

2. The serration behaviors of the alloy are closely related to the strain rate. At a certain deformation temperature, the serration curves first start with the type A ($10^{-3}$ s$^{-1}$), then transfers to the mixed type A + B ($10^{-2}$ s$^{-1}$) and eventually to type D ($10^{-1}$ s$^{-1}$) with the increasing strain rates from $10^{-3}$ s$^{-1}$ to $10^{-1}$ s$^{-1}$. The critical strain ($\varepsilon_c$) increases with the increasing strain rate and the decreasing temperature, which suggests the occurrence of the normal PLC effect in the Ni-based superalloy.

3. The $m + \beta$ value was calculated to be 2.51, which suggests that the serrated flow behavior is related to the interplay between the substitutional solute atoms (especially the atom Cr) and the movable dislocations. The activation energy for the serration of the alloy is calculated to be 103.386 kJ mol$^{-1}$. In addition, the precipitation phase can influence the serrated flow behavior by affecting the interplay among mobile dislocations, forest dislocations, and substitutional solute atoms.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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