Portevin–Le Chatelier Effect in Nimonic 263 Superalloy

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Abstract The Portevin–Le Chatelier (PLC) effect in the Nimonic 263 superalloy was investigated by tensile test at a wide temperature ranges from 293 to 1033 K and strain rates between 0.1 and $6.25 \times 10^{-6} \text{ s}^{-1}$. Simple binary alloys Ni-0.4C, Ni-24Cr and Ni-5(8)Mo were also tested in order to identify which elements were responsible for the PLC effect in the Nimonic 263 alloy. The results demonstrated that for Nimonic 263 alloy, PLC effect occurred at certain temperatures and low strain rates. Normal PLC effect exhibiting type-A and -(A + B) serrations was attributed to the enhanced solute diffusion with increasing temperature, while inverse PLC effect with type-C serration was caused by unlocking process. The activation energy for the normal PLC effect was calculated to be 68 kJ/mol, and diffusion of substitutional solutes such as Cr and Mo was identified to be responsible for the PLC effect. In comparison with the PLC effect in simple binary alloys, solute atmospheres formed by different kinds of atoms in Nimonic 263 alloy work more effectively, increasing locking strength and corresponding mean stress drop magnitude.

KEY WORDS: PLC effect; Dynamic strain aging; Nickel-base alloys; Solute atom

1 Introduction

In order to improve power plant efficiency and reduce CO₂ emissions, advanced ultra-supercritical (A-USC) power plant with higher efficiency has been developed for many years. Comparing with the current supercritical coal-fired power plant operating at steam temperatures about 600 °C and 30 MPa, A-USC power plants are designed to run at higher steam temperatures up to 700–760 °C with pressures of 35 MPa [1–3]. To achieve such advanced steam conditions, many critical components such as boiler tubing and reheater pipes require the materials with improved creep strength and better corrosion resistance. The

replacement of the ferritic alloys used in USC by nickel-base superalloys seems to be essential, and a number of nickel-base superalloys such as CCA 617 [4], Inconel 740 [1, 2] and Nimonic 263 [5] have been chosen as the candidate materials for A-USC applications. Nimonic 263 is a wrought nickel-base superalloy that is widely used in gas turbines due to its better creep strength and oxidation resistance. The phase stability and the mechanical properties of this alloy have been investigated in detail [5–7]. However, limited information is available in the literature on the PLC (serrated flow) in this alloy.

The PLC effect, which manifests itself as serrations on the stress–strain curves, often occurs in many interstitial and substitutional solid solutions within a certain range of temperature and strain rate [8–10]. During the process of plastic deformation, such unstable plastic flow leads to inhomogeneous strain localization that is commonly referred to as PLC bands [11, 12]. These PLC bands result in rippled surfaces of the samples and are usually undesirable in industrial processes. Moreover, dynamic strain aging

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(DSA) has a large effect on the mechanical properties. Some studies [13, 14] have reported that yield strength or ultimate tensile strength shows a distinct peak or plateau within the temperature range of the PLC effect.

In general, the PLC effect is attributed to DSA, which is associated with interaction between the mobile dislocations and diffusing solute atoms [15, 16]. According to the dependence of the critical strain (ε_c , which is required for the onset of serrated flow) on the temperature and strain rate, there exist two types of the PLC effect termed as normal PLC effect and inverse PLC effect, respectively. The normal PLC effect is often observed at low temperatures, in which ε_c is found to increase with increasing strain rate and decreasing temperature, while at high temperatures, the inverse PLC effect exists in which the critical strain increases with decreasing strain rate or increasing temperature. Based on the concept firstly proposed by Cottrell [15] and then developed by Van den Beukel [17] and McCormick [18], solute atoms responsible for the PLC effect can be identified through estimating the activation energy of diffusion by

$$\varepsilon_c^{m+\beta} = \left(\frac{C_s}{\alpha C_0} \right)^{3/2} \frac{\dot{\varepsilon} k T b \exp\left(\frac{Q_m}{kT}\right)}{3 L N K U_m D_0}, \quad (1)$$

where C_s is the local solute concentration at the dislocation required for locking, C_0 is the matrix solute concentration of the alloy, $\alpha \approx 3$, L is the average distance between arresting obstacles, U_m is the solute–dislocation binding energy, D_0 is the diffusion frequency factor, Q_m is the activation energy for solute migration, and K , N , m , β are constants. By using this method, Hayes [19] proposed that the normal PLC effect is the result of carbon atmosphere pinning of dislocation at low temperatures and the draining of carbon due to a precipitation reaction is the main reason for the inverse PLC effect. Other researchers [20, 21] reported that the PLC effect at low temperatures (below 825 K) is caused by interstitial carbon atoms, whereas the inverse PLC effect at high temperature is caused by substitutional solutes. Therefore, there still exist some controversies about which atoms are responsible for the PLC effect in a complex superalloy system. Moreover, it should be mentioned that $m + \beta$ is related to the vacancy concentration C_v and the mobile dislocation density ρ_m by the relationship [22]

$$C_v = K \varepsilon^m, \quad (2)$$

$$\rho = N \varepsilon^\beta, \quad (3)$$

C_v and ρ_m increase with increasing strain, and the exponent $m + \beta$ should be positive. Therefore, the method to calculate activation energy of atom diffusion cannot be used in the case at high temperatures because the value of $m + \beta$ becomes negative, just as demonstrated in the reference [20]. That is to

say, the method proposed by McCormick [18] can be only applied to the process dominated by atom diffusion and locking the mobile dislocations at low temperatures. In fact, studies by Fu *et al.* [23] have also confirmed that the inverse PLC effect at high temperature is determined by the pinning strength instead of diffusion of the specific solute atoms. It is therefore questionable to calculate the activation energy to identify which solute atoms are responsible for the inverse PLC effect at high temperature.

The aim of the present study was to investigate the influences of the strain rate and the temperature on the PLC effect in Nimonic 263 alloy. By calculating the activation energy for the normal PLC effect at low temperature, solute atoms responsible for the PLC effect are estimated. Then in comparison with the temperature regime for PLC effect occurrence and mean stress drop magnitude in simple binary alloys such as Ni-0.4C, Ni-24Cr and Ni-5(8)Mo (in at%), solute atmospheres formed by different kind of atoms were proposed to interpret the PLC effect in the complex superalloy system.

2 Experimental

The nominal chemical compositions (in at%) of Nimonic 263 alloy used in this investigation are 12Co, 22Cr, 4Mo, 2.3Ti, 1.2Al, 0.3C, 0.5Mn, 0.8Si and the balance Ni. Ingots were prepared by vacuum induction melting. After homogenization, the ingots were hot forged at 1100 °C to 30 mm (diam.) bar, followed by a solution annealing at 1150 °C for 3 h, and then water quenched to room temperature. Binary alloys Ni-0.4C, Ni-24Cr and Ni-5Mo (at%) were designed according to atomic ratio of (Ni + Co) with C, Cr and Mo in Nimonic 263. In order to study the effect of solute concentration on the PLC effect, Ni-8Mo (at%) alloy was also tested. Ni-0.4C alloy after forging was heat-treated at 1000 °C for 30 min, and Ni-24Cr, Ni-5Mo and Ni-8Mo alloys were heat-treated at 1150 °C for 3 h in order to obtain a similar grain size.

The specimens with a gage section of 3 mm in diameter and 20 mm in length were used in tensile tests. The tensile tests were carried out in air at temperatures ranging from 293 to 1033 K and the strain rates between 0.1 and $6.25 \times 10^{-6} \text{ s}^{-1}$. Before the start of the tensile test, specimens were heated to the desired test temperatures in a split-type, three-zone electric resistance heating furnace and held for 10 min to stabilize and homogenize the temperature over the gage section. In comparison with PLC effect in Nimonic 263 alloy, Ni-0.4C, Ni-24Cr and Ni-5(8)Mo alloys were tested in a wide temperature regime at a strain rate $4 \times 10^{-4} \text{ s}^{-1}$. The samples for the microstructure observation by scanning electron microscopy (SEM) were prepared using conventional metallographic procedures and chemically etched by Kalling solution.

3 Results

3.1 Microstructure Characterization of Nimonic 263 Alloy

Figure 1 shows the microstructures of the alloys after solution annealing followed by water quenching. The grain size is determined to be about 200 μm from the optical micrograph (Fig. 1a). There are some twins formed within the grain. The SEM micrograph (Fig. 1b) reveals that some irregular γ' particles homogeneously precipitate in the γ matrix.

3.2 Serrated Flow in Nimonic 263 Alloy

Figure 2 shows the typical true stress–strain curves for the specimens tested in the temperature range of 293–1033 K at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$. At this strain rate, smooth stress–strain curves are observed at temperatures below 373 K. Serrations appear on the stress–strain curves between 473 and 1033 K. At higher strain rates, the regime over which serrated flow is observed is shifted to higher temperatures. The different types of serrations are identified as types A, B and C according to the classification scheme in the literature [24, 25] and are summarized on the serration map shown in Fig. 3. The trend of the serration appears to change from type A to type C with increasing the temperature or decreasing the strain rate. For example, at the lower strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ as shown in Fig. 2b, type-A serrations which are characterized by an abrupt rise in stress followed by a drop to or below the general level of the stress–strain curves were observed at 473 K. Type B serrations fluctuate around the general level of the stress–strain curve occurred between 723 and 773 K.

With the temperature increasing to 923 K, type-C serrations dropped below the general level of the stress–strain curve.

The critical strain ε_c for the onset of serration flow is evaluated from the true stress–strain curves as the minimum value of plastic strain at which a perceptible stress drop of 0.4 MPa occurs. A trend is noticed in the variation of ε_c with temperature at a constant strain rate of $4 \times 10^{-4} \text{ s}^{-1}$, as shown in Fig. 4. For a given strain rate, it is found that ε_c decreases with increasing temperature investigated up to 723 K and the normal PLC effect takes place; thereafter, ε_c increases with temperature, and the inverse PLC effect is seen.

Strain-rate sensitivity, m , of flow stress, σ , at a given T and ε was evaluated using $\sigma - \varepsilon$ data from tensile tests at constant cross-head speeds through the relation [26]

$$S = \frac{\log(\sigma_2/\sigma_1)}{\log(\dot{\varepsilon}_2/\dot{\varepsilon}_1)} \bigg|_{\varepsilon, T},$$

where σ_1 and σ_2 are the flow stresses at strain rates $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$, respectively. Values of m at $\varepsilon = 5\%$ level were determined with $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ as 4×10^{-4} and $8 \times 10^{-3} \text{ s}^{-1}$, respectively. Variation of S with different temperatures is shown in Fig. 5, where S values are positive below 373 K. When serrations are observed on the stress–strain curves, S values become negative.

Figure 6 shows the distributions of the stress drop magnitude $\Delta\sigma$ at a constant strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ under different test temperatures, which correspond to the different serration types. It can be seen that at the low temperature of 473 K, type-A serration exhibits power-law function in the distribution of the $\Delta\sigma$, while a transition occurs from the power-law function to a single peak statistical function with increasing the test temperature.

Fig. 1 The microstructures of the alloys after solution annealing followed by water quenching: **a** optical micrograph, **b** SEM micrograph

Fig. 2 **a** Typical true stress–true strain curves over the temperature range of 293–1033 K at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ and **b** various types of serrated flow observed in the true stress–true strain curves

Fig. 3 Regimes of occurrence of serrated flow for Nimonic 263 superalloy

Fig. 5 Variation of strain-rate sensitivity with temperature at a true plastic strain of 5%

Fig. 4 Variation of critical strain for the onset of serrations with the temperature at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$

Similar to the temperature dependence, the distributions of the stress drop magnitude are changed from the power-law function to the single peak statistical function with decreasing the strain rate under certain temperatures; this will not be discussed further in the present paper. The temperature dependence of the mean stress drop is shown in Fig. 7. It can be seen that the mean stress drop increases with increasing temperature up to about 800 K and then decreases.

3.3 Serrated flow in Ni-0.4C, Ni-24Cr and Ni-5(8)Mo alloys

Figure 8 exhibits the true stress–strain curves for the simple binary alloys such as Ni-0.4C, Ni-24Cr, Ni-5Mo and Ni-8Mo under the same conditions as that in Nimonic 263

Fig. 6 The distribution of stress drop magnitude at the typical test temperatures and a strain rate $4 \times 10^{-4} \text{ s}^{-1}$: **a** 473 K, **b** 773 K, **c** 1033 K

Fig. 7 The change of mean stress drop with temperatures at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ in Nimonic 263 alloy

alloy. It can be seen that the PLC effect of Ni-0.4C alloy is visible between the temperatures of 293 and 473 K, and it disappears over the temperature of 573 K, and the PLC effect occurs in Ni-24Cr alloy between the temperatures of 573 and 923 K. Although the PLC effect does not appear in Ni-5Mo alloy over the whole temperature range, it occurs between 573 and 723 K with increasing the content of Mo atom to 8 at%.

4 Discussion

The present study demonstrates that Nimonic 263 alloy in a quenched state showed a serrated flow or the so-called PLC effect when they are tensile tested under certain temperature and strain-rate regimes. It is evident that variation of serration type in the PLC effect is controlled by DSA and therefore mainly influenced by concentration and diffusion rate of solute atoms, mobile and forest dislocation densities. It should be noted that variation of serration type with temperature or strain rate indicates the transition of

the underlying mechanisms, namely from locking process at low temperature (or high strain rate) to unlocking process at high temperature (or low strain rate) [23]. For Nimonic 263 alloy, type-A serrations which show upward serrations above the general level of stress-strain curves are observed below the test temperature of 573 K, while type-C serrations exhibiting downward serrations appear at about 923 K. Between 573 and 923 K, the types of serration correspond to a transition state, namely type-(A + B) or -B serrations. Similar results are also observed in alloy 625 [14] and Inconel 718SPF [20]. In fact, type-A serrations at low temperatures are dominated by the locking process of diffusing solute atoms, whereas type-C serrations at high temperatures may be due to unlocking of initially aged dislocations. That is to say, macroscopic serrations are mainly determined by microscopic aging process. As recently proposed by Fu et al. [23] in Al-Mg alloy, at low temperatures, dislocation is in the unlocked state and the dominant process is diffusion of solute atoms to the dislocation, exerting an additional stress on the mobile dislocations and leading to upward serrations on the flow stress curves. At high temperatures, dislocations are in the locked state before the start of plastic deformation. When the movement of the dislocations is activated under the applied stress, downward serrations are produced.

In addition to serration types, another two parameters, critical strain ε_c and stress drop magnitude $\Delta\sigma$, are more critical in the investigation of PLC effect. The critical strain signifies whether it is easy to lock dislocations by solute atoms or break off from solute atmospheres for the locked dislocations. By using McCormick's method [18], a plot of $\ln(\varepsilon_c^{m+\beta}/T)$ versus $1/T$ for a given strain rate yields activation energy below the temperature of 723 K through the relationship of $Q_m = \text{slope} \times R$, where R is the gas constant. However, this method needs evaluation of $(m + \beta)$ to compute $\varepsilon_c^{m+\beta}$ at temperature regime where critical strain versus $1/T$ shows a normal behavior. $m + \beta$ can be obtained by measurement of the strain-rate dependence of ε_c by the following equation [18]

Fig. 8 True stress–true strain curves of simple binary alloys over the temperature range of 293–1033 K at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$: **a** Ni-0.4C, **b** Ni-24Cr, **c** Ni-5Mo, **d** Ni-8Mo

Fig. 9 Calculated method of the activation energy for the normal PLC effect in Nimonic 263 alloy: **a** the plot of $\ln \dot{\epsilon}$ versus $\ln \epsilon_c$; **b** the plot of $\ln [\dot{\epsilon}_c^{(m+\beta)}/T]$ versus $1/T$

$$\dot{\epsilon}_c^{m+\beta} = K \dot{\epsilon} \exp(Q/RT). \quad (4)$$

The typical plot of $\ln \dot{\epsilon}$ versus $\ln \epsilon_c$ at a temperature of 573 K is shown in Fig. 9a with a slope of 2.3 for $m + \beta$ value. By using the value of $(m + \beta)$, the activation energy for the locking serration from the plot of $\ln (\dot{\epsilon}_c^{m+\beta}/T)$

against $1/T$ is calculated to be about 68 kJ/mol, just as shown in Fig. 9b. The activation energy for the onset of serration has been taken as the solute migration energy and is important to identify which atom is responsible for the PLC effect. It seems that the activation energy in the present study is very close to the activation energy for bulk

diffusion of interstitial C atom in nickel [27]. However, most experimental results [28–30] indicated that instead of bulk diffusion, pipe diffusion with lower activation energy is the dominant mechanism during dynamic strain aging process. And the activation energy for pipe diffusion is in the range (40–70)% of the activation energy for lattice diffusion. Therefore, it is likely that the possible solutes responsible for the PLC effect of Nimonic 263 alloy at low temperature are substitutional atoms, such as Cr and Mo, rather than C atom. The activation energy for lattice diffusion of Cr or Mo in Ni has been reported as 272 [31] and 213 kJ [27], respectively, and the average activation energy of diffusion by pipe diffusion is about 0.5 times the activation energy for lattice diffusion. Additionally, this view is further corroborated by the observed $(m + \beta) = 2.3$. In general, in the PLC effect involving substitutional solutes, values of $(m + \beta)$ are in the range 2–3, whereas for the PLC effect due to interstitial solutes, values of $(m + \beta)$ are in the range 0.5–1.

In order to confirm this postulation, simple binary alloys in Ni-0.4C, Ni-24Cr and Ni-5Mo were tested under the same condition. The temperature range yielding the PLC effect and serration types found in Ni-24Cr alloy are in excellent agreement with that for the PLC phenomenon in Nimonic 263 alloy. Hence, it seems that the major substitutional solute of Cr in Nimonic 263 alloy is responsible for the PLC effect. However, if only Cr atom was predicted to be responsible for the PLC effect, mean stress drop for PLC effect in Ni-24Cr should be approximately equal to that in Nimonic 263 alloy. But mean stress drop of Ni-24Cr alloy is largely low in comparison with that of Nimonic 263 alloy, just as shown in Fig. 10. In order to explain this discrepancy, the following speculations should be pointed

Fig. 10 Comparison of mean stress drop variation with increasing the temperature at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ for Nimonic 263 and Ni-24Cr alloys

out: (1) an effective solute atmosphere requires a minimum solute concentration $C_{\min}$ available for diffusing toward mobile dislocations and locking them and (2) the atom with lower solute content than $C_{\min}$ may be involved to form a solute atmosphere with the dominant element and work together with it in dynamic strain aging process. For the minimum solute concentration required for locking, it has been reported in many references [14, 23, 32]. In this case, the serrated flow is not obviously observed in Ni-5Mo alloy, which does not mean that Mo atoms have no effect on the dynamic strain aging process. Moreover, from the test results of Ni-8Mo alloys, the serrated flow phenomenon was found, which indicates that Mo atoms make contributions to the dynamic strain aging. Only when the solute concentration of Mo reaches a certain critical content, the macroscopic PLC effect or serrated flow revealed on the tensile curves. Thus, in Nimonic 263 alloy, it is likely that Cr atoms combined with other substitutional solute atoms constitute more effective solute atmospheres. In these solute atmospheres, Cr may act as dominant element during dynamic strain aging process. Other substitutional solute atoms diffuse along mobile dislocation line, form multi-atomic solute atmospheres with Cr atoms, and work together with them, resulting in the increase of locking strength and corresponding stress drop magnitude. As for the disappearance of serrations below the temperature of 473 K in Nimonic 263 alloy, it may be associated with a carbide formation and carbon atmosphere cannot be formed due to its depletion.

For the inverse PLC effect occurring in the high temperature regime, it is believed that the diffusion rates are high enough for dislocations to be aged from the start of deformation and type-C serrations appear to be due to breakaway of the aged dislocations. The onset of serrations may be determined by solute concentration around mobile dislocations, the aging time of solute diffusing to dislocation and dislocation density. Shankar *et al.* [14] thought that if a solute concentration required for the formation of effective locking is insufficient, it may not lead to the flow serrations; meanwhile, if the aging time for the concentration buildup is not sufficient due to the application of a high strain rate, the solute concentration cannot also be formed.

The statistics of stress drop seems to correlate with the PLC band types and the underlying mechanism for the PLC effect. When temperature is increased at a given strain rate, or when the imposed strain rate is decreased at a given temperature, the distribution of stress drop changes from power-law function to single peak function, and correspondingly, the types of serrations evolve from type A to type C. An asymmetrical peak distribution corresponds to the intermediate band types (A + B) or B. The nature of the stress drop denotes the magnitude of locking or

unlocking stress, which is exerted on the mobile dislocations by solute atmospheres. Distribution of the stress drops showing power-law function possibly results from the locking process, which produces tiny stress serrations at low temperature, while it will change gradually into the peak distribution with increasing the temperature since additional stress increases with raising diffusivity of solute atoms.

Negative strain-rate sensitivity, m , of flow stress, shown in the regime of the serrated flow, is in agreement with the behavior in other alloy systems exhibiting the PLC effect [10]. Penning [33] pointed out that a negative strain-rate dependence of the flow stress is a necessary condition for the occurrence of the serrated flow, and negative m values result in strain localization and macroscopic inhomogeneous deformation. In the microscopic inspect, it is thought that aging of mobile dislocations by solute atoms takes place when mobile dislocations are prevented from moving continuously by obstacles [18]. The combined action of dynamic aging and dislocation interactions is considered essential for the occurrence of negative strain-rate sensitivities.

5 Conclusions

- (1) The PLC effect occurred at certain temperatures and low strain rates in Nimonic 263 alloy. At a constant strain rate of $4 \times 10^{-4} \text{ s}^{-1}$, the PLC effect was found to occur in the temperature range of 473–1033 K. The trend of the serration appears to change from type A to type C with increasing temperature. Type-A serrations were observed at the temperatures of 473 K, and beyond 773 K, type-C serrations were seen.
- (2) The activation energy for the normal PLC effect was obtained to be about 68 kJ/mol. Substitutional solute atoms such as Cr and Mo were identified to be responsible for the PLC.
- (3) In comparison with the PLC effect in simple binary alloys, multi-atomic solute atmospheres formed in Nimonic 263 alloy work together more effectively, increasing locking strength and mean stress drop magnitude.

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