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On the mechanism of serrated deformation in aged Inconel 718

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Abstract

The serrated deformation of Inconel 718 alloy aged at 725°C for various lengths of time has been studied in the temperature range from 200 to 575°C and at constant strain rates in the range of 10^{-2} to 5×10^{-6} s^{-1}. It was observed that serrated flow in the alloy depends on the interaction mechanism of dislocations with precipitates. Normal serrated flow was observed when deformation occurred by dislocations cutting through precipitates. However, when the Orowan looping mechanism was operative in the overaged material, normal serrated flow was observed only when the strain rate was high or the test temperature was low. In addition, the critical strain to serration was observed to increase with an increase in inter-particle spacing. This observation is inconsistent with the models proposed by Hayes et al. and McCormick. Therefore, a new mechanism, which considers the specific interaction mechanism of a moving dislocation with precipitates, has been proposed. © 1997 Elsevier Science S.A.

Keywords: Serrated deformation; Inconel 718; Orowan looping mechanism

1. Introduction

The occurrence of the Portevin–Le Chatelier (PLC) effect has been extensively studied in many alloy systems [1–3]. A 'normal' behaviour which shows a positive dependence of the critical onset strain on strain rate and a negative dependence on temperature were often observed in many dilute solid solutions and its mechanism is reasonably well understood. However, in concentrated solid solutions, an 'inverse' behaviour which shows a negative dependence of the critical strain with strain rate and a positive on temperature were also found, but its mechanism remains to be understood. This inverse behaviour was reported mostly in Al–Mg alloys, and has also been observed in Ni base alloys [4–8].

The occurrence of the PLC effect in nickel based alloys was attributed to the presence of interstitial solutes such as C and N [4–7]. The activation energy responsible for it further suggests that the serrated flow involves dislocation core diffusion rather than the bulk diffusion by which the carbon atmosphere interacts with moving dislocations [4,7]. The inverse serration behaviour was also found in nickel based alloys when precipitates were present. This has been modeled by Hayes et al. [7,8]. They have proposed that the strain delay for the onset of serration in aged Waspalloy depends on the reaction between the $Ni_3(Al,Ti)$ precipitate and carbon in the atmospheres on the dislocations arrested at precipitates. Carbon, draining off the dislocation lines by pipe diffusion down a carbon activity gradient to the $Ni_3(Al,Ti)$ precipitates, allows the dislocation line to advance without having to pull away from the atmosphere. The rate controlling process for the delay in the onset of serration is the reaction between the carbon and the $Ni_3(Al,Ti)$ precipitate. This reaction is rapid initially and then slows progressively with strain as the sinks approach saturation.

In this communication, the dependence of serrated flow on interparticle spacing in preaged nickel base Inconel 718 has been studied. Some results inconsistent with Hayes' model were observed, for which an explanation has been provided.

2. Experimental methods

The chemical composition (wt.%) of Inconel 718 used in this study was 0.03/C, 19.24/Fe, 52.37/Ni, 18.24/Cr,

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0.52/Al, 0.97/Ti, 0.07/Mo, 4.98/(Nb + Ta), 0.007/Mn, 0.007/S, 0.30/Si, 0.04/Cu. A 2.54 mm thick sheet of the alloy was cold-rolled to a thickness of 1.4 mm and machined into flat samples. The samples were first solution treated at 1020°C for 4 h, and then aged at 725°C for various lengths of time. The heat treated samples were grounded on SiC paper up to 600 grade to obtain a gauge dimension of 1.3 × 5.3 × 25.4 mm. The microstructures of heat treated samples were examined in a JEOL 2000FX analytical TEM/STEM. The precipitate particle size was measured by using enlarged micrographs on a Leitz Image Analyzer. Tensile tests were carried out in the temperature range of 200–575°C at strain rates ranging from 10^{-2} to 5×10^{-6} s^{-1} on an Instron testing machine.

3. Results

3.1. Dependence of serrated flow on γ'' precipitate particle size

The major strengthening precipitates in Inconel 718 are ordered disc shaped BCT γ''-Ni_3(Nb, Fe) and ordered spherical FCC γ'-Ni_3(Al, Ti, Nb) [9–11]. The latter provides only a minor contribution to the overall strength (10–20%) [11]. For this reason, often only γ''-precipitates are considered whenever precipitation-strengthening is considered in this material, which is also the case in the present study.

The variation in the 0.2% yield stress of specimens aged at 725°C with aging time and particle size at the testing temperature of 300 and 425°C is shown in Figs. 1 and 2, respectively. The peak strength at 300°C was observed to occur at a particle size of about 23 nm

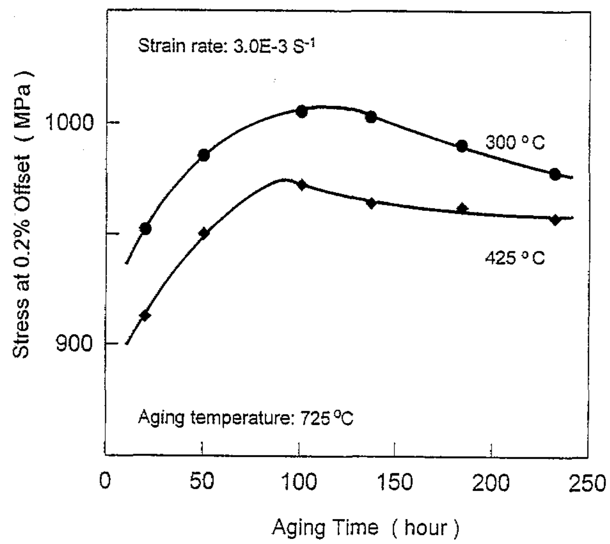

Fig. 1. Variation in 0.2% yield stress of material aged at 725°C with aging time at the testing temperature of 300 and 425°C.

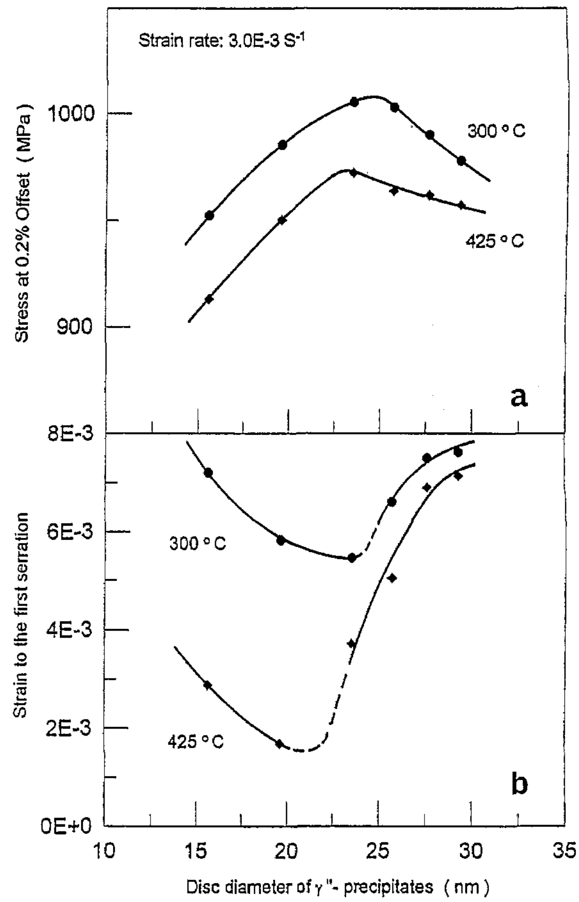

Fig. 2. Dependence of 0.2% yield stress (a) and strain to the first serration (b) on disc diameter of γ'' precipitates at the testing temperature of 300 and 425°C.

(after more than 100 h, Fig. 1), while at 425°C the peak strength occurred at a somewhat lower particle size (< 100 h, in Fig. 1). Serrated flow was observed at these temperatures and the critical strain to the onset of serrations is also plotted in Fig. 2. It is seen that the critical strain to serrations at both the temperatures first decreases to a minimum, then increases with an increase in particle size. The particle size that delineates the two regions is nearly the same as the γ'' particle size that divides the underaged and the overaged regions. The increase in critical strain to the onset of the first serration with particle size is an indication of delayed serration behavior.

3.2. Dependence of critical strain on strain rate

Fig. 3 shows the dependence of critical strain to the onset of serrations on strain rate. When the material is underaged ($d_{\gamma''} \approx 19$ nm), normal serrated flow is observed. That is, the critical strain increases with an increase in strain rate. In the overaged condition, however, the curve ($d_{\gamma''} \approx 25$ nm) consists of both a normal and an inverse serrated flow. In this case, the critical

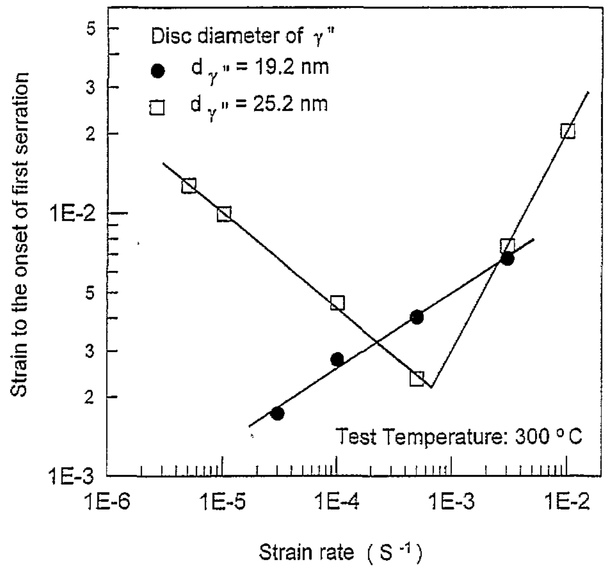

Fig. 3. Dependence of critical strain on strain rate.

strain is observed to first decrease with an increase in strain rate in the low strain rate region, and then increases in the higher strain rate region.

3.3. Dependence of serrated flow on temperature:

The effect of test temperature on the critical strain to serration in both the underaged and the overaged material is shown in Fig. 4, where critical strain to serration, ε_c, is plotted against $1/T$. It is seen that in the underaged condition ($d_{\gamma''} \approx 19$ nm) the value of ε_c decreases with the temperature but in the overaged condition ($d_{\gamma''} \approx 25$ nm) it first increases, then decreases. It is likely that a transition also occurs in the underaged condition, but at a higher temperature.

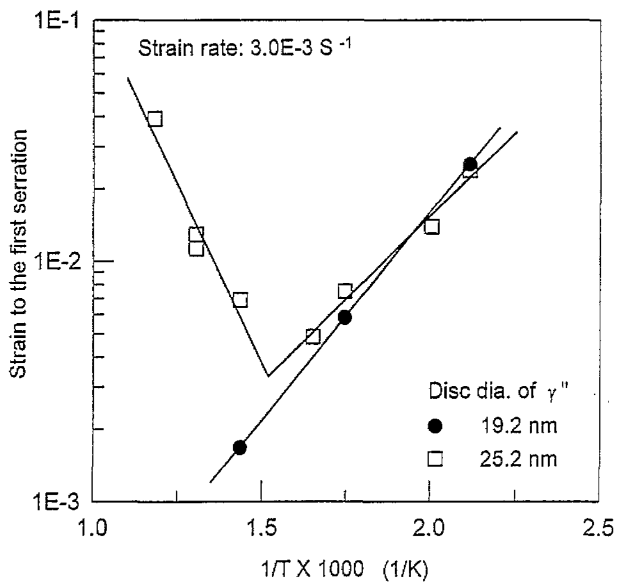

Fig. 4. Dependence of critical strain on test temperature.

According to Ham and Jaffrey [12], the relation between the critical strain and constant test strain rate can be expressed as:

$$\dot{\varepsilon}_c = B\varepsilon_c^m \exp(-E_m/kT) \quad (1)$$

where B and m are constants, and E_m is the effective activation energy for the thermally activated process responsible for serrated flow. In this expression, m can be determined from the slope of the log E_c vs log E_c plot, and E_m can be determined directly from a log E_c versus $1/T$ curve at a constant. Using this relationship, the value of m and E_m were calculated to be 3.44 and 2.75 eV, respectively, for the underaged material where the diameter of γ'' was 19 nm. In the overaged material where the diameter of γ'' was 25 nm and a normal serration behaviour was observed, the value of m and E_m were 1.2 and 0.75 eV, respectively.

4. Discussion

In the underaged condition ($d_{\gamma''} \approx 19$ nm), the observed value of m is beyond the range that characterizes serrated flow controlled by interstitial elements. However, the value of m may not be the criterion for determining the mechanism that controls serrated flow. As was observed in Waspalloy [7], the slope of log ε_c vs. log $\dot{\varepsilon}_c$ plot increases with an increase in temperature. Accordingly, the value of E_m determined from Eq. (1) should be invalid. In the overaged condition ($d_{\gamma''} \approx 25$ nm) which exhibits normal serration behavior, the observed value of m seems to be consistent with the carbon atmosphere anchoring mechanism as found by Nakada and Keh in Ni–C alloys [4]. In this case, the determined value of activation energy (0.75 eV) is also in a reasonable agreement with the values reported by other researchers ($0.52 \sim 0.68$ eV) [4,7]. This suggests that the serration behaviour in Inconel 718, like that in Waspalloy, may involve interstitial elements instead of substitutional elements as was suggested by Hayes [8] to explain the disappearance of serrations in this alloy.

In the precipitate-strengthened material, the critical strain to serrations is often related to the average spacing between particles. The particle spacing, L, as a function of volume fraction, f, and mean planar cross-section of a particle in the slip plane, r_s, is given by $L = [(\pi/f)^{1/2}\text{-}2]r_s$, where, $r_s = (2/3)^{1/2}\ r$, r being the particle radius [13]. It was found that with an increase in the aging time of Inconel 718 at 725°C, the volume fraction of precipitates in the material can increase rapidly to a level (about 14–17%) that did not change during further increases in aging time [11]. This suggests that f in Inconel 718 can be assumed to be constant. As a result, L can be considered to be linearly proportional to particle size. That is, the dependence of serration behavior on particle size, as presented in Fig. 2, is also a reflection of the dependence of the serration behavior on interparticle spacing.

The effect of interparticle spacing on serration behavior can be described by the following expression based on the static strain aging model suggested by Sleeswyk [14], and developed in detail by McCormick [15].

$$\varepsilon_c^m = BL^{-1}\dot{\varepsilon}_c \exp(Q_m/kT) \quad (2)$$

here B is a constant, $\dot{\varepsilon}_c$ is the plastic strain rate, Q_m is the effective energy for the thermally activated process responsible for serrated flow, k and T have their usual meanings, and m is a constant equal to $0.5 \sim 1.0$ for nickel base alloys. The validity of the dependence of ε_c on L has been experimentally verified, especially, in materials in which substitutional elements are responsible for serrated flow [16,17].

In nickel base precipitate-strengthened alloys where interstitial elements are responsible for serrations, the dependence of ε_c on L has not been studied, although Hayes and Hayes [7] have speculated about the possible dependence. They propose that, when the material is less effectively aged, the precipitates are further apart. Consequently, the atmospheres must diffuse down a longer pipe to drain the dislocation line and more carbon must be drained. Thus, at a given strain rate there is a smaller value of critical strain needed for the onset of serrated flow for the material with widely spaced precipitates than for the optimum aged material. According to this concept, a continuous decrease in the critical strain to the onset of serrations should be observed in the entire range of interparticle spacing. In the present study, this is observed only when the material was in the underaged condition (Fig. 2).

Since the serration behavior in the present study was observed to vary with particle size in a way that is nearly similar to the dependence of yield strength on particle size, the effect of the dependence of yield strength on particle size may have to be considered. It is widely accepted that after a short aging time, when the particles are small and coherent with the matrix, dislocations can cut through them [13]. As the aging time increases, the size of particles and the spacing between them are increased, the shearing process becomes more difficult and less probable and the Orowan bow-out mechanism prevails. The peak yield stress delineates the dislocation cutting mechanism and the Orowan looping mechanism. It has been also observed that the critical size of precipitates that determines the peak strength also decreases with an increase in temperature [18]. For example, this critical size in Inconel 718 is found to be about 26 nm at room temperature, and about 22 nm at 600°C [18]. This observation is consistent with the results of the present study. As seen in Fig. 2, the peak strength is observed to occur at a smaller particle size at the higher test temperature. In addition, a similar variation was observed in critical strain to serrations at these two temperatures.

Considering the similarity between the dependence of yield stress and strain to onset of serrations on particle size, the following is proposed to explain serrated flow in Inconel 718.

4.1. Underaged material

In this condition, a moving dislocation can cut through the coherent precipitates [13]. The interstitial atmospheres, which have possibly segregated to the bowed out segment of the dislocation core when it is arrested at precipitates, may diffuse to the portion of the same dislocation which is within the precipitates. This is due to the carbon activity gradient which exists along the line of the dislocation that drives the diffusion [7]. The majority of precipitates in the material are γ'', which has a lattice misfit of $+2.86\%$ with the surrounding matrix [19] so that the portion of dislocation within a γ'' precipitate may have larger interstitial space at its core than the rest of the dislocation. The interstitial atmospheres, therefore, will be also more stable when segregated at the dislocation core within precipitates. When the interstitial elements are segregated to the portion of a dislocation within a precipitate, its subsequent effect on a moving dislocation can be assumed to occur in the following two ways:

(1) If interstitial elements continue their association with the moving dislocation, the entire moving dislocation is not free of interstitial atmospheres, and should be also retarded by them. As a result, a normal PLC effect, irrespective of the existence of precipitates, should be expected. When normal serrated flow is present, the dependence of critical strain to the onset of serrations, ε_c, on the average interparticle spacing, L, can be described by Eq. (2), which predicts that the critical strain is inversely proportional to the interparticle spacing. This prediction is consistent with the results presented in Fig. 2.

(2) Interstitial elements may interact with the precipitates or may be left behind with the precipitates as the entire dislocation escapes from the pinning particles. This suggests that a delayed onset of serrations could occur if a reaction is possible and its rate is fast enough. Unfortunately, this phenomenon was not observed at the test conditions used in the present studies. However, if a higher temperature or slower strain rate is used, it might be possible for the interstitial atmosphere to bond to the carbide-forming elements in the precipitates as was proposed by Hayes and Hayes [7].

4.2. Overaged material

In this condition, a direct interaction of interstitial elements with precipitates, as it occurs during the cutting process, may be unlikely, since a moving dislocation will form a loop around particles as the dislocation by-passes them. This loop can serve as a pocket for receiving the interstitial atmospheres that may have diffused down the pipe line from the bowed-out segment of the moving dislocation. This is probably because the portion of dislocation confronting the precipitates will expand its length rapidly while the bowed-out segment is arrested at precipitates. The newly created dislocation line would be free of segregation of interstitial elements. The concentration difference between the two will drive the interstitial elements to migrate to the loop region. Although the diffusion process may not be able to drain all the interstitial elements off the bowed-out segment of dislocation, it can reduce its concentration to the level where a serration becomes invisible. Based on this concept, the occurrence of serrations should depend on a balance between the segregation rate of interstitial elements to the bowed-out segment of a dislocation and diffusion rate of the interstitial elements to the loop region around a precipitate. As a result, a delayed serration can be observed if the test temperature is high or the strain rate is low. In both situations, the diffusion of interstitial elements to the loop region is possible. On the other hand, normal serrated flow can be observed instead, if the test temperature is low or strain rate is high. In this situation, serrations will occur if interstitial elements can be segregated to the bowed-out part of a dislocation. This suggestion is in good agreement with the experimental results obtained in the present study. As shown in Figs. 3 and 4, the inverse dependence of the critical strain on strain rate and inverse of test temperature occurs when the strain rate is lower and the temperature is higher, while a normal dependence has been observed when the strain rate is higher and the test temperature is lower.

The positive dependence of ε_c, on L in overaged material ($d_{\gamma''} \approx 25$ nm) can be caused by the following two reasons:

4.2.1. The waiting time of a moving dislocation at particles

McCormick [15] suggested that the average dislocation velocity, $\bar{v}$, can be expressed in terms of an arrest or waiting time at the obstacles, t_w, and a time of flight through the lattice to the next obstacle, t_f, as follows,

$$\bar{v} = L/(t_w + t_f) \tag{3}$$

where L is the distance between obstacles. In particle-strengthened material, L is usually assumed to be equal to interparticle spacing. It is assumed that t_w is determined largely by the obstacle strength, while t_f depends on viscous drag forces acting on the moving dislocation. It is suggested that $\bar{v}$ is determined primarily by t_w. Therefore, t_w has been expressed as,

$$t_w \approx L/\bar{v} = L\rho b/\dot{\varepsilon}_c \tag{4}$$

where ρ is the mobile dislocation density and b is the Burgers vector. By equating t_g with the aging time [20], Eq. (2) was obtained [15].

This treatment would be applicable when a mobile dislocation cuts the precipitates. In this situation, a bigger particle size (longer interparticle spacing) requires a larger stress to cut the precipitates, so that the waiting time will be longer and the value of L will be larger. Therefore, in accordance with Eq. (2) the strain to serrations will be smaller. This is consistent with our results.

However, when the Orowan mechanism is operating, the shear strength, τ, is inversely proportional to L, that is, $\tau \approx Gb/2L$. As McCormick assumed, if t_w is proportional to the particle strength, then, $t_w \propto 1/L$. This is inverse of the situation in the underaged material where $t_w \propto L$. In addition, the flight time, t_f, in the overaged material, which is directly proportional to the interobstacle spacing, may not be small enough to be neglected, as was done in the derivation of Eq. (4). Therefore, Eqs. (4) and (2) would not be applicable to the overaged material. A modified Eq. (2) is not available, however, qualitatively it is suggested that since in the overaged material $t_w \propto 1/L$, a longer delay in the onset of serrations should be expected as the interparticle spacing or the particle size increases. This is also consistent with the observations on overaged material (25 nm) presented in Fig. 2.

4.2.2. Diffusion of interstitial elements to the dislocation loop

When the waiting time is longer than the time required for the interstitial atmospheres to segregate to the dislocation core region, the serrations may still not be able to occur. This is because the interstitial elements may diffuse from the bowed out segment to the dislocation loop, which remains with the particle, and may not have any significant effect on further motion of the entire dislocation. This will cause an extra delay in the onset of the first serration. This might be the reason why in Fig. 2(b) the increase in ε_c in the right branch of the curve for 425°C (for overaged materials) is more pronounced than that of the 300°C-curve. The serrations are delayed to a higher degree as the diffusion to the loop is more effective at 425°C. This argument is also consistent with the temperature dependence of ε_c for overaged material ($d_{\gamma''} \approx 25$ nm) as presented in Fig. 4. It is observed that at temperatures 425°C and above the value of ε_c increases with an increase in temperature and at temperatures below 350°C it decreases. This means that at lower temperatures (right branch of the plot in Fig. 4) the serrations are controlled by the segregation of interstitial elements to the dislocation core. However, at higher temperatures (left branch of the plot in Fig. 4) the segregated elements can diffuse to the dislocation loops around precipitate particles, and would cause an additional delay in the onset of serrations. The additional delay would be reduced at larger particles (i.e. longer interparticle spacing) as segregated interstials have to diffuse a longer distance to the dislocation loops around precipitates. For a given particle size and inter particle spacing in the overaged material the value of ε_c, thus, reflects which controlling process is dominant and how rapidly the two processes occur.

5. Conclusions

(1) When dislocations can cut through precipitates, the critical strain to serrations was observed to decrease with an increase in interparticle spacing (L). In the overaged condition, however, the critical strain was found to increase with an increase in L. This observation is inconsistent with the models proposed by McCormick [15] and Hayes and Hayes [7] who predicted that the critical strain should decrease with an increase in L regardless of the dislocation-precipitate interaction mechanism.

(2) The occurrence of serrations in the pre-aged Inconel 718 involves the segregation and depletion of interstitial elements to and from dislocation cores. The segregation process depends on the waiting time of dislocations at particles, t_w. In the underaged condition, t_w increases with an increase in L, while it decreases with an increase in L in the overaged condition. This explains why the critical strain depends on L differently in underaged and overaged material. The depletion process depends on how a moving dislocation interacts with precipitates. When a dislocation can cut through precipitates, depletion may be unlikely since the interstitial elements are still associated with the dislocation core even though they might diffuse to the precipitates. When the Orowan mechanism is dominant, interstitial elements can diffuse to the dislocation loop formed around precipitates. When this occurs, an unusual serration behavior might be expected.

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References

[1] J.M. Robinson and M.P. Shaw, *Int. Mater. Rev.*, *39* (1994) 113.
[2] Viewpoint Set on Propagative Plastic Instabilities, *Scr. Metall. Mater.*, *29* (1993) 1147.
[3] Y. Brechet and Y. Estrin, *Acta Metall. Mater.*, *43* (1995) 995.
[4] Y. Nakada and A.S. Keh, *Acta Metall.*, *18* (1970) 437.
[5] J.S. Blakemore, *Metall. Trans.*, *1* (1970) 1281.
[6] R.A. Mulford and U.F. Kocks, *Acta Metall.*, *27* (1979) 1125.
[7] R.H. Hayes and W.C. Hayes, *Acta Metall.*, *30* (1982) 1295.
[8] R.W. Hayes, *Acta Metall.*, *31* (1983) 365.
[9] J.F. Barker, in H.L. Eiselstein, *Advances in the Technology of Stainless Steels and Related Alloys*, *STP 369*, ASTM, Philadelphia, PA, 1965, p. 78.
[10] P.S. Kotval, *Trans. TMS-AIME*, *242* (1968) 1764.
[11] Y. Han, P. Deb and M.C. Chaturvedi, *Met. Sci.*, *16* (1982) 555.
[12] R.K. Ham and G. Jaffrey, *Philos. Mag.*, *44* (1953) 829.
[13] L.M. Brown and R.H. Ham, in A. Kelly and R.B. Nicholson (eds.), *Strengthening Mechanisms in Crystals*, Elsevier, New York, 1971, p. 7.
[14] A.W. Sleeswyk, *Acta Metall.*, *6* (1958) 598.
[15] P.G. McCormick, *Acta Metall.*, *20* (1972) 351.
[16] D.J. Lloyd, D.W. Chung and M.C. Chaturvedi, *Acta Metall.*, *23* (1975) 93.
[17] D.W. Chung, M.C. Chaturvedi and D.J. Lloyd, *Acta Metall.*, *24* (1976) 227.
[18] M.C. Chaturvedi and Y. Han, *Mater. Sci. Eng.*, *89* (1987) 25.
[19] C.T. Sims and W.C. Hagel, *The Superalloys*, Wiley, New York, 1972, p. 199.
[20] J. Friedel, *Dislocations*, Pergamon Press, 1964, p. 405.