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Plastic flow instabilities of $L1_2$ Ni_3Al alloys at intermediate temperatures

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The serrated plastic flow of $L1_2$ Ni_3Al alloys at intermediate temperatures was investigated using tensile tests. The effects of temperature, strain rate and composition were examined. The serrated plastic flow accompanied by the lowest (negative) strain-rate sensitivity was observed most strongly at 673 K and at a strain rate of 3.2×10^{-4} s^{-1}. The serrated plastic flow became more significant as the alloy departed from a stoichiometric composition. The static strain aging at 673 K resulted in a reduced flow strength. The activation energy of the serrated plastic flow was estimated to be about 66 kJ/mol, which suggests that it is smaller than that for lattice diffusion of solutes in $L1_2$ lattices. The serrated plastic flow behavior of the Ni_3Al alloys was compared with that of the $L1_2$ Co_3Ti and Ni_3(Si,Ti) alloys, and is qualitatively explained on the basis of the dynamics of solutes in the core of dissociated screw dislocations. © *2004 Kluwer Academic Publishers*

1. Introduction

Many ordered intermetallic alloys as well as some disordered alloys exhibit an anomalous increase of flow strength with increasing temperature at intermediate temperatures [1–3]. Some of such strength anomalies observed in ordered alloys have been attributed to dynamic strain aging (DSA) [4], by which most of strength anomalies observed in disordered alloys have been explained [5–9]. When solutes dynamically interact with moving dislocations, and have their strongest interaction at intermediate temperatures and strain rates, DSC takes place, resulting in strength increases and serrated plastic flow curves. The $L1_2$ Co_3Ti and Ni_3(Si,Ti) alloys that display the strength anomaly [10, 11] have been recently reported to show serrated plastic flow at intermediate temperatures [12, 13]. The observed plastic flow instability has been suggested to be understood as DSA in which solutes in the core of dissociated screw dislocations (i.e., in the inelastic field of dislocations) play an important role and affect the dynamics of dislocations [12, 13].

The Ni_3Al alloys have many similarities to the Co_3Ti and Ni_3(Si,Ti) alloys, and for example actually exhibit an anomalous increase of flow strength with increasing temperature at intermediate temperatures [14–18]. In this study, the serrated plastic flow behavior of the Ni_3Al alloys is shown to take place at intermediate temperatures in tension tests. In addition, the temperature, strain rate and composition dependence of the serrated plastic flow behavior of the Ni_3Al alloys is presented. Static strain aging (SSA) and strain-rate change tests were also performed. Based on these results, a possible mechanism of the serrated plastic flow behavior, i.e., dynamic strain aging (DSA) in the Ni_3Al alloys is suggested, and comparisons are made not only with disordered alloys but also with Co_3Ti and Ni_3(Si,Ti) alloys.

2. Experimental procedure

500 wt.ppm boron-doped Ni_3Al alloys with nominal compositions of 24, 23 and 22.5 at.%Al were used in this study. Raw materials used in this study were 99.9 wt% nickel, 99.99 wt% aluminum and 99.5 wt% boron. Alloy button ingots with dimensions of 15 mm $\times$ 25 mm $\times$ 50 mm were prepared by arc melting in an argon gas atmosphere on a copper hearth using a non-consumable tungsten electrode. A homogenization heat treatment was conducted in vacuum at 1323 K for 2 days, followed by furnace cooling. Plates sliced from these buttons, about 4 mm in thickness, were cold-rolled to 2.5 mm in air and then annealed in vacuum at 1273 K for 5 h. After annealing, the plates were also cold-rolled to about 1 mm in air. Tensile specimens with 1 mm $\times$ 2 mm cross-section and 10 mm gage length were cut from these sheet materials using an electro-discharge machine. These tensile specimens were recrystallized in vacuum at high temperature to obtain microstructures with desired grain sizes. To obtain identical grain sizes in the three alloy compositions, the 24 at.%Al, 23 at.%Al and 22.5 at.%Al alloys, were finally recrystallized at 1173 K for 1 h, 1273 K for 2 h and 1323 K for 2 h, respectively. The obtained grain sizes were about 10 μm.

For tensile tests and mechanical evaluation for DSA, almost the same procedure with that conducted on the $L1_2$ Co_3Ti and Ni_3(Si, Ti) alloys [10, 11] was taken in this study. Yield stress, maximum stress amplitude, strain-rate sensitivity (SRS), static aging (SSA) and activation energy for the present DSA were evaluated e.g., in terms of alloy composition, temperature, strain rate and so on. The strain-rate values that led to the most significant serrated plastic flow were plotted against $1/T$, that is, an Arrhenius plot was produced, based on an equation,

$$\dot{\varepsilon} = \mathrm{A}\ \exp(-Q/RT) \tag{2}$$

where $\dot{\varepsilon}$ is strain rate resulting in the maximum stress amplitude (or the largest serration), Q is activation energy for the present phenomenon, R is the gas constant and T is the temperature.

3. Results

Fig. 1 shows that the microstructures of the 24 at.%Al and 23 at.%Al alloys consisted of equiaxed $L1_2$ grains,

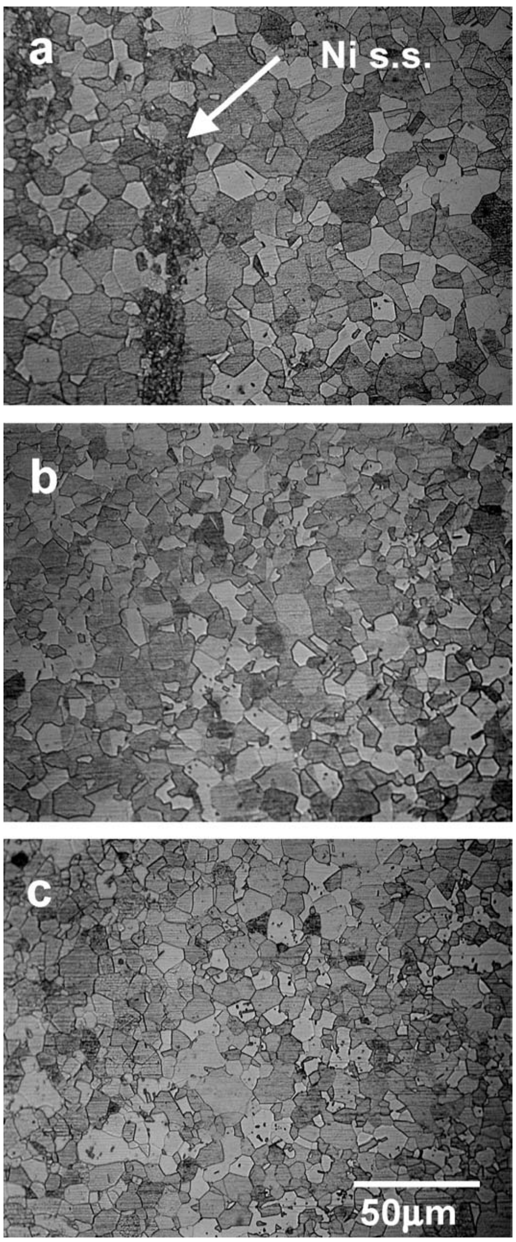

Figure 1 Optical microstructures of the (a) 22.5 at.%Al, (b) 23 at.%Al and (c) 24 at.%Al alloys, respectively. Note that denoted Ni s.s. means Ni sold solution.

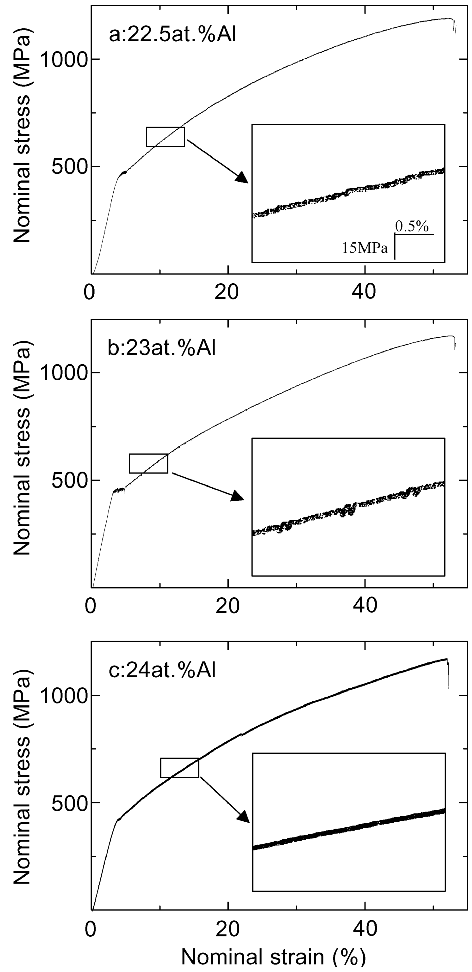

Figure 2 Nominal stress-strain curves of the (a) 22.5 at.%Al, (b) 23 at.%Al and (c) 24 at.%Al alloys, respectively. The tests were conducted at 673 K and at a strain rate of $3.2 \times 10^{-4}\ \mathrm{s}^{-1}$.

accompanied with a moderate number of twin boundaries, while the microstructure of the 22.5 at.%Al alloy contained Ni solid solutions in $L1_2$ grains.

Fig. 2 shows nominal (i.e., engineering) stress-strain curves of the 24 at.%Al, 23 at.%Al and 22.5 at.%Al alloys which were deformed at 673 K and at a strain rate of $3.2 \times 10^{-4}\ \mathrm{s}^{-1}$. In the 24 at.%Al and 23 at.%Al alloys consisting of $L1_2$ mono-phase grains, the serrated plastic flow becomes more significant with deviation from the stoichiometric composition, but in the 22.5 at.%Al alloy containing Ni solid solution, the serrated plastic flow becomes less significant in spite of the greater deviation from the stoichiometric composition.

Detailed features relating to the serrated plastic flow observed in the 23 at.%Al alloy are shown in Fig. 3. The yield stress begins to increase from 573 K and still tends to increase even at the highest temperature (i.e., 773 K) used in this study (Fig. 3a). On the other hand, the maximum stress amplitude begins to increase from 573 K with increasing temperature and makes a peak (∼13 MPa) at 673 K, followed by a decrease at higher temperatures (Fig. 3b). On the other hand, the SRS begins to decrease from 573 K with increasing temperature and makes a minimum (−11 MPa) at 673 K, followed by an increase at higher temperatures (Fig. 3c). The SRS values between 623 and 723 K were negative: the yield stress decreases with increasing strain rate at

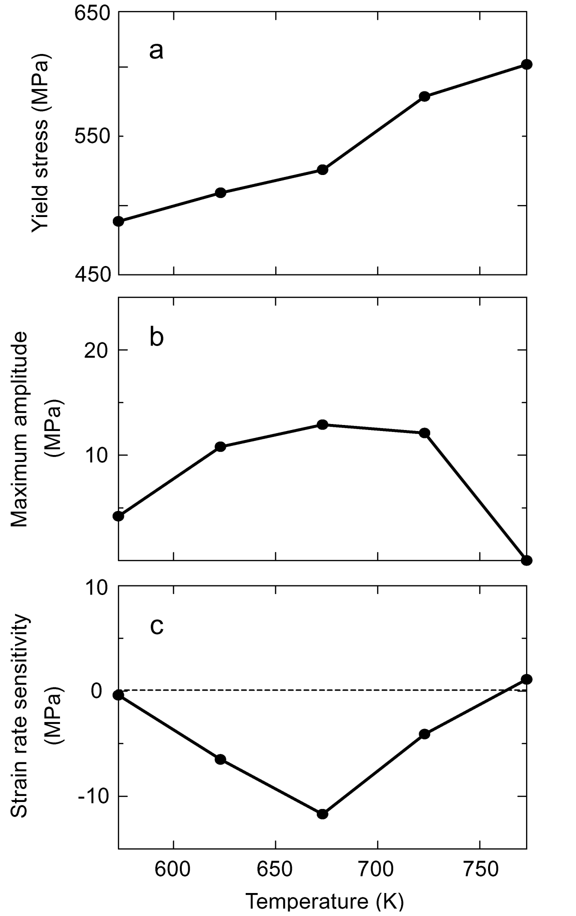

Figure 3 Changes of (a) yield stress, (b) maximum stress amplitude and (c) SRS for the 23 at.%Al alloy. Date of (a) and (b) were taken at a strain rate of 3.2×10^{-4} s^{-1}, while date of (c) were calculated from the two strain rates 3.2×10^{-5} s^{-1} and 3.2×10^{-4} s^{-1}.

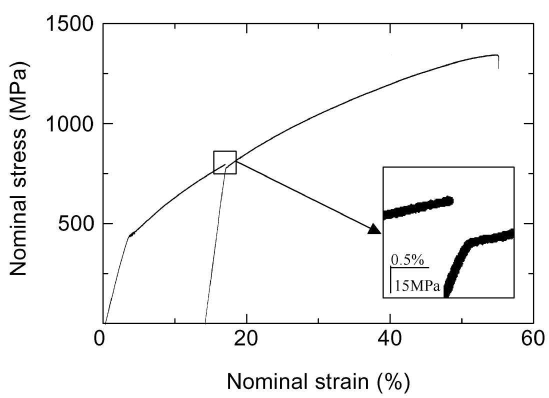

Figure 4 Static strain aging experiment on the 23 at.%Al alloy at 673 K at a strain rate of 3.2×10^{-4} s^{-1}. 2 h was taken as a duration time until reloading.

these temperatures. It is thus noted that the temperature region where the DSA, characterized by the maximum stress amplitude and the SRS, takes place is lower and narrower than that where the positive temperature dependence of the yield stress, i.e., the strength anomaly, takes place.

Fig. 4 shows the result of the SSA test on the 23 at.%Al alloy performed at 673 K and for a duration time of 2 h until reloading. Strength did not increase and instead unexpected strength decrease was observed. Also, the yielding behavior was not observed upon reloading, in contrast to the case of first loading.

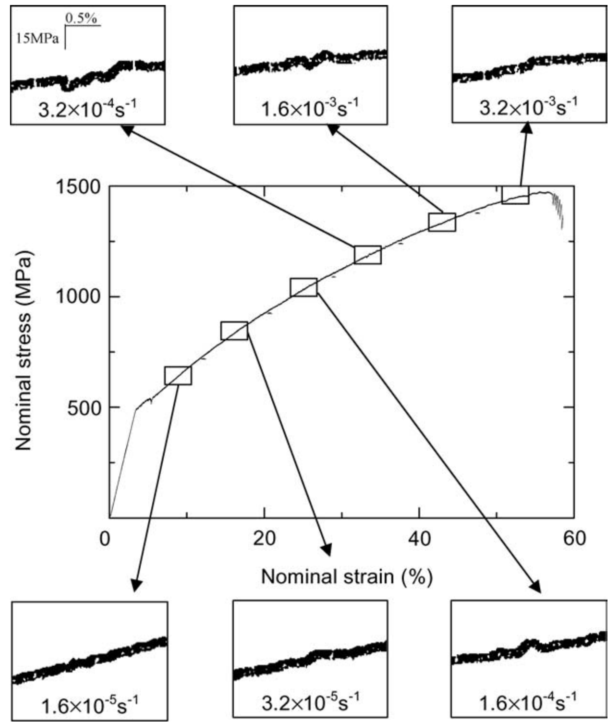

Figure 5 Strain rate change test on the 23 at.%Al alloy deformed at 723 K. The strain rate was successively changed from 1.6×10^{-5} s^{-1} to 3.2×10^{-3} s^{-1}.

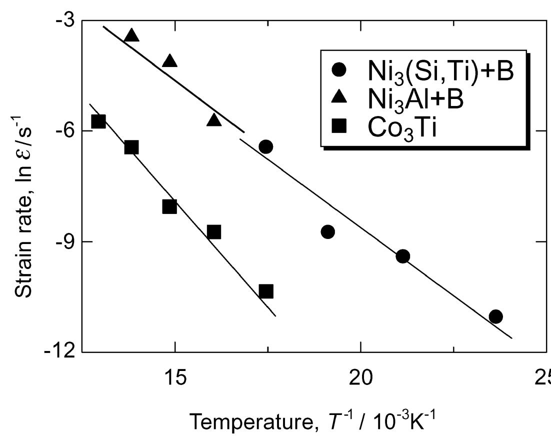

Figure 6 An Arrhenius plot of the strain-rate value that displayed the most significant serrated plastic flows at each test temperature. Data for the Co_3Ti [12] and Ni_3(Si,Ti) [13] alloys were included for reference.

The result of the strain-rate change test performed at 723 K is shown in Fig. 5. In this case, the most significant serrated plastic flow was identified to take place at a strain rate of 3.2×10^{-4} s^{-1}. The strain rate that displayed the most significant serrated plastic flow was plotted against $1/T$, as shown in Fig. 6. Consequently, a value of 66 kJ/mol was calculated as the activation energy for the present DSA phenomenon.

4. Discussion

The present results indicate that the DSA in the Ni_3Al alloys is affected not only by temperature and strain rate but also by alloy composition. Furthermore, many results for the DSA observed in the Ni_3Al alloys are quite similar to those observed in the Co_3Ti [12] and Ni_3(Si,Ti) [13] alloys, as shown in Table I. For example,

TABLE I Comparison in the DSA behavior among the Ni_3Al, Co_3Ti [12] and $Ni_3(Si, Ti)$ [13] alloys

Alloy	Co_3Ti [12]	$Ni_3(Si,Ti)$ [13]	Ni_3Al
Stoichiometry	21–23 at.%	20.5 at.%	22.5–24 at.%
Temperature	673 K	473 K	673 K
Serrated flow	Strong	Moderate	Moderate
SRS	−15.1 MPa	−3.9 MPa	−11.7 MPa
SSA, $\Delta\sigma$	Soften	Soften	Soften
Activation energy	84 kJ/mol	57 kJ/mol	66 kJ/mol

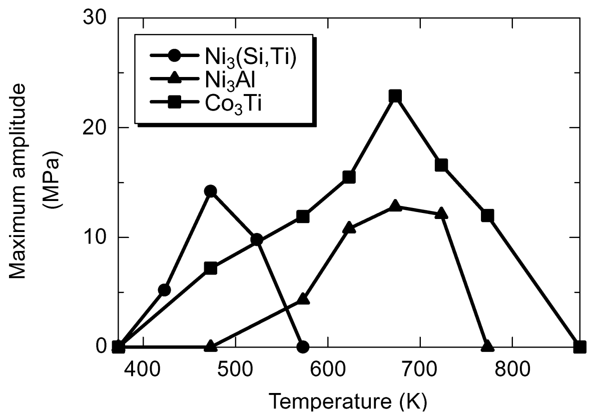

Figure 7 Change of the maximum stress amplitude for the Ni_3Al alloy with temperature. Data for the Co_3Ti [12] and $Ni_3(Si,Ti)$ [13] alloys were included for reference.

(1) The serrated plastic flow became more significant with departing from a just-stoichiometric composition. (2) The serrated plastic flow took place in lower and narrower temperature region than the positive temperature dependence of the yield strength, as shown in Fig. 7. (3) The most significant serrated plastic flow took place at intermediate temperatures and also at intermediate strain rates. (4) Negative values of the SRS were observed in the temperature region where the serrated plastic flow took place. (5) The SSA did not strengthen but soften the Ni_3Al alloys. (6) The evaluated activation energy was a low value (less than 100 kJ/mol) which is not considered as the activation energy for lattice diffusion of solutes.

Some experimental results in the Ni_3Al alloys as well as the Co_3Ti [12] and $Ni_3(Si, Ti)$ [13] alloys may be consistent with the classic DSA mechanisms based on interaction of solutes with moving dislocations [5–9]. For example, the terms (1), (3) and (4) mentioned in the previous section are consistent with the classic DSA mechanisms. The largest serrated flow is expected to take place when the diffusion of solute corresponds to the velocity of moving dislocations. Excess (or anti-site) Ni atoms might be considered to behave as solute atoms. Furthermore, negative values of the SRS have been obtained in the temperature region where the significant serrated plastic flow took place. Such features have been actually reported for the DSA behavior of many intermetallic alloys [19–21].

Some results (e.g., terms (5) and (6)) in the Ni_3Al alloys as well as the Co_3Ti [12] and $Ni_3(Si,Ti)$ [13] alloys are inconsistent with the classic DSA mechanisms [5–9]. For example, strengthening was not observed but softening was observed for static aging. This result implies that during annealing at intermediate temperatures, recovery is superior to formation of solute atmosphere halting dislocation motion. Secondly, the estimated activation energy (66 kJ/mol) appears to be far smaller than that for diffusion of solutes (Ni or Al solutes) in the $L1_2$ lattice, and to correspond to the activation energy for diffusion of solutes in the dislocation core although such data are not available for the Ni_3Al alloy. This fact means that solutes contained in the dislocation core dominate the present phenomenon. Thirdly, it has been reported by TEM observation for deformed Ni_3Al alloys [22, 23] that the dislocations activated in the temperature regime where the positive temperature dependence of the yield strength takes place, and therefore the DSA takes place, are long and straight with screw character. In the classic DSA mechanisms, it has been implicitly suggested that solutes do not interact with screw dislocations but prefer to interact with edge dislocations due to their large elastic interaction [24]. Considering these inconsistencies, the serrated plastic flow observed in the Ni_3Al alloys is not satisfactory explained by the classic DSA mechanisms.

The strength anomaly of the Ni_3Al alloys took place up to the temperature region beyond 770 K (Fig. 3), as has also been repeatedly reported in the previous studies [14–18]. However, the temperature region showing the serrated plastic flow was lower and more confined than that showing the strength anomaly. Therefore, the serrated plastic flow and the related phenomena observed in the Ni_3Al alloys are not explained by the KW mechanism [25–28], although the KW locks by a thermal process operate as an underlying mechanism up to the peak temperature of strength anomaly, and result in low strain-rate sensitivity of the yield strength at this whole temperatures. It is possible that the decomposed screw dislocations can be immobilized by redistribution of solutes within the dislocation core [12, 13]. At temperatures far below the peak temperature of the strength anomaly, solutes can modify and then immobilize the core of decomposed screw dislocations, resulting in jerky motion of dislocations, i.e., serrated plastic flow. Corresponding to this process, more negative values of the strain-rate sensitivity are expected in the low temperature regime (i.e., the DSA regime) than in the high temperature regime (i.e., the KW locks regime).

In the DSA operating in $L1_2$ ordered alloys such as Ni_3Al, $Ni_3(Si, Ti)$ and Co_3Ti, it is suggested that solutes interact with decomposed screw dislocations in their inelastic strain field, modifying the core structure of decomposed screw dislocations, as shown in Fig. 8. Consequently, the activation energy for the DSA in $L1_2$ ordered alloys should be that for the core diffusion of solutes in dislocations while the activation energy at for the DSA in disordered alloys should be that for the lattice (bulk) diffusion of solutes.

As possible solutes affecting the core structure of decomposed screw dislocations, interstitials such as boron, gaseous elements or other unknown impurities are not likely to operate in this study. Reasons are that

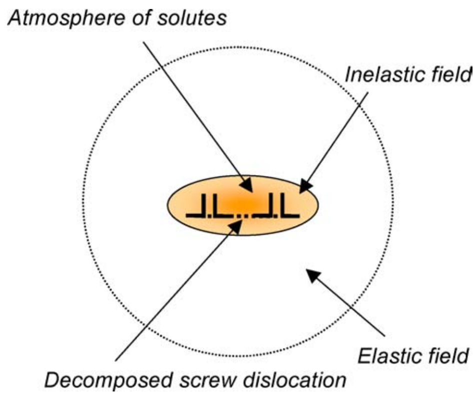

Figure 8 Interaction of solutes with a dislocation core in $L1_2$ ordered intermetallic alloys.

the observed DSA is primarily sensitive to alloy stoichiometry, i.e., constituent elements, and also that in the previous study for the Ni_3(Si, Ti) alloys with and without boron, the observed DSA has been little sensitive to boron doping [13]. Alternatively, it is suggested that excess Ni atoms, or depleted Al atoms modify the core structure of decomposed screw dislocations, immobilizing the dislocations.

5. Conclusions

The effects of temperature, strain rate and alloy stoichiometry on the DSA of the $L1_2$ Ni_3Al alloys were investigated using tensile tests. The following conclusions were obtained from the present study.

1. At a strain rate of 3.2×10^{-4} s^{-1} the serrated plastic flow was observed most strongly at 673 K. Also, the maximum stress amplitude and the lowest (negative) strain-rate sensitivity were observed at 673 K.
2. The serrated plastic flow became more significant as the alloy departed from a stoichiometric composition. Also, the static strain aging at 673 K resulted in a reduced flow strength.
3. The activation energy for the serrated plastic flow was estimated to be about 66 kJ/mol, which suggests that it corresponds to the activation energy for the diffusion of solutes in the dislocation core.
4. The serrated plastic flow behavior of the Ni_3Al alloys was not attributed to the classic DSA mechanisms, instead, to the dynamic behavior of the solutes in the core structure of dissociated screw dislocation.

References

1. T. SUZUKI, Y. MISHIMA and S. MIURA, *ISIJ Int.* **29** (1989) 29.
2. D. CAILLARD and A. COURET, in "Dislocations in Solids," Vol. 10, edited by F. R. N. Nabarro and M. S. Duesberry (Elsevier, Amsterdam, 1996) Chapt. 50, p. 69.
3. D. CAILLARD, *Mater. Sci. Eng.* A **319–321** (2001) 74.
4. E. O. HALL, "Yield Point Phenomena in Metals and Alloys" (Plenum, New York, 1970).
5. I. POTEZ, G. LAPASSET and L. P. KUBIN, *Scripta Metal. Mater.* **26** (1992) 84.
6. J. F. DINHUT, T. BONOU and P. MOINE, *Acta Metal.* **24** (1976) 445.
7. R. LERF and D. G. MORRIS, *Acta Metal. Mater.* **42** (1994) 1091.
8. H. MABUCHI, K. HIRUKAWA, K. KATAYAMA, H. TSUDA and Y. NAKAYAMA, *Scripta Metal.* **24** (1990) 1553.
9. E. P. GEORGE, D. P. POPE, C. L. FU and J. H. SCHNEIBEL, *ISIJ Int.* **31** (1991) 1063.
10. T. TAKASUGI, S. HIRAKAWA, O. IZUMI, S. ONO and S. WATANABE, *Acta Metall.* **35** (1987) 2015.
11. T. TAKASUGI, S. WATANABE, O. IZUMI and H. K. FATHALLA, *ibid.* **37** (1989) 3425.
12. T. TAKASUGI, H. HONJO, Y. KANENO and H. INOUE, *ibid.* **50** (2002) 847.
13. H. HONJO, Y. KANENO, H. INOUE and T. TAKASUGI, *J. Mater. Res.* **17** (2002) 705.
14. P. VEYSSIERE and G. SAADA, in "Dislocations in Solids," Vol. 10, edited by F. R. N. Nabarro and M. S. Duesbery (Amsterdam, North-Holland, 1996) Chapt. 53, p. 254.
15. D. P. POPE and S. S. EZZ, *Int. Mater. Rev.* **29** (1984) 136.
16. N. S. STOLOFF, *ibid.* **34** (1989) 153.
17. M. YAMAGUCHI and Y. UMAKOSHI, *Prog. Mater. Sci.* **34** (1990) 1.
18. T. SUZUKI, Y. MISHIMA and S. MIURA, *ISIJ Int.* **29** (1989) 1.
19. F. POPILLE, L. KUBIN, J. DOUIN and S. NAKA, *Scr. Metall.* **34** (1996) 977.
20. M. DEMURA and T. HIRANO, *Phil. Mag. Letts.* **75** (1997) 143.
21. P. H. THORNTON, R. G. DAVIES and T. L. JOHNSTON, *Metall. Trans.* **1** (1970) 207.
22. Y. Q. SUN and P. M. HAZZLEDINE, *Phil. Mag.* A **58** (1988) 263.
23. P. VEYSSIERE and J. DOUIN, in "Intermetallic Compounds—Principles," edited by J. H. Westbrook and R. L. Fleischer (John Wiley and Sons, New York, 1995) p. 519.
24. A. H. COTTRELL, "Dislocations and Plastic Flow in Crystals" (Clarendon Press, Oxford, 1953).
25. B. H. KEAR and H. G. F. WILSDORF, *Trans. Metal. Soc. AIME* **224** (1962) 382.
26. S. TAKEUCHI and E. KURAMOTO, *Acta Metall.* **21** (1973) 415.
27. V. PAIDAR, D. P. POPE and V. VITEK, *ibid.* **32** (1984) 435.
28. M. H. YOO, *Scripta Metall.* **20** (1986) 915.

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