

# Dislocation arrangement in the plastic zone of propagating cracks in nickel

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The structure of the plastic zone ahead of a crack has been studied in nickel using the *in situ* transmission electron microscopy technique. The cracks formed in the thin foils were shear cracks of mode III type. For tensile axis orientations near  $[1\ 1\ 1]$ , the plastic zone was in the form of a thin ribbon consisting of an inverse pile-up of partial dislocations. This structure changed to a broad array of perfect dislocations for the orientations near  $[00\ 1]$ . The observation of partial dislocations in nickel may be considered anomalous because of its relatively high stacking fault energy. The orientation dependence of the splitting is discussed in terms of various mechanisms such as impurity, stress-induced separation of partials, and mutual interaction between the partials. It is concluded that the stress-induced nucleation and separation of the partials are responsible for the orientation dependence but other factors such as the mutual interaction of the partials must be considered to explain the observed width of the splitting.

## 1. Introduction

The distribution of dislocations in the plastic zone ahead of a crack tip has been studied recently in various metals during *in situ* tensile deformation in an electron microscope. In stainless steel [1] and copper [2], the plastic zone is co-planar with the crack, the dislocations in the plastic zone are in the form of an inverse pile-up, and they are split into partial dislocations. These cracks are identified as antiplane strain shear cracks of mode III type. On the other hand, in aluminium [3] and in bcc metals such as molybdenum and tungsten [4], the plastic zone is somewhat broad and consists of several dislocation arrays, some of which are branched out from the main pile-up. The dislocations in the plastic zone are perfect dislocations which cross-slip easily. This behaviour reflects the differences in the stacking fault energy of these metals.

In the present study, the structure of the plastic zone in nickel has been studied using the *in situ* TEM fracture technique. Nickel is considered to be a metal of intermediate stacking fault energy [5, 6]. It is shown that the dislocations in the

plastic zone in nickel can be either split or unsplit depending on the orientation of the stress axis.

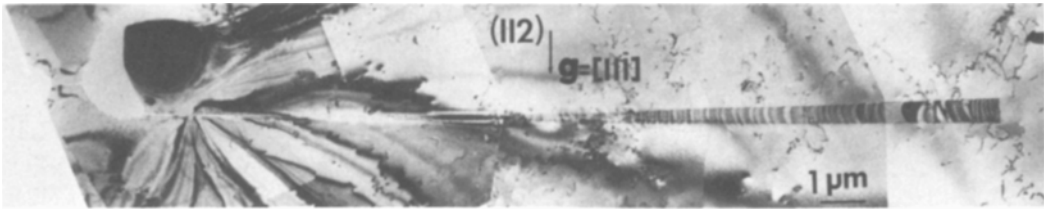
## 2. Experiments

Electron microscope specimens of nickel were prepared from polycrystalline sheets of commercial purity and zone-refined single crystal rods of high purity. These specimens were electropolished in a Tenupol unit with a solution of 70% ethyl alcohol, 10% butyl cellsolve, 8% perchloric acid, and 12% water at  $-5^{\circ}\text{C}$ . The specimens were deformed in a bending holder [7] which allows normal operation of the tilting stage of a Hitachi HU 200E electron microscope. Since the specimens were polished from one side, the tensile stress could be applied to the thinned area near the polishing hole.

## 3. Results

When tensile stress was applied to the thinned area initially, dislocations were observed to move from the thick area toward the edge of the hole. After a considerable amount of slip, cracks were initiated at the edge of the hole and propagated into the specimen. During crack propagation, many dislo-

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**Figure 1** Electron micrograph showing a shear crack and its plastic zone in polycrystalline nickel specimen of commercial purity. The plastic zone consists of an inverse pile-up of partial dislocations with stacking faults between the partials. A dislocation-free zone can be seen near the crack tip.

cations were generated at the crack tip and moved into the specimen to form a plastic zone. Two distinct distributions of dislocations in the plastic zones associated with the cracks were observed. The plastic zone shown in Fig. 1 appears as a thin ribbon and consists of a number of partial dislocations with stacking fault fringes. This micrograph was taken from a polycrystalline specimen. The foil surface was close to  $(1\ 1\ 2)$ . From stereoscopic observations the crack was found to be close to mode III type and the plane of the plastic zone was identified as  $(1\ 1\ 1)$ . The dislocation density was very high near the crack tip and decreased gradually away from the crack. It was noted that the area immediately ahead of the crack tip was dislocation-free. This is the inverse pile-up of dislocations first predicted by Bilby *et al.* [8] and recently discussed by Chang and Ohr [9, 10]. These features of the plastic zone are very similar to those observed in metals of low stacking fault energy, namely copper [2] and stainless steel [1].

The second type of plastic zone observed was not in the form of a thin ribbon. As shown in Fig. 2, the plastic zone was rather broad and consisted of many perfect dislocations. The micrograph was also taken from a polycrystalline specimen with a diffraction vector of  $[\bar{1}\ 1\ \bar{1}]$ . The foil surface was close to  $(1\ 1\ 0)$ . The crack plane as well as the plane of the plastic zone was identified

as  $(1\ 1\ \bar{1})$ . The direction of the dislocations was roughly parallel to the projected direction of  $[1\ 0\ 1]$ , which was identified as the direction of the Burgers vector. This again indicated that the dislocations were pure screws and the crack was of mode III type. Plastic zones of similar nature have been observed in aluminium [3] and bcc metals [4].

The relationship between the dislocation splitting and the orientation of the tensile axis was analysed. In many cases, the tensile axis was approximately perpendicular to the crack direction with the deformation occurring on the primary slip system. However, in some cases slip was activated on the secondary slip system. Analyses were made only for the cases where the primary slip system was activated. The results are summarized in Fig. 3. The open circles indicate the tensile axis orientations where the dislocations within the plastic zone are split into partials and stacking faults are observed. The orientations where the plastic zone consists of perfect dislocations are indicated by the closed circles. The stereographic triangle can be divided into two areas representing the orientations of splitting and non-splitting.

Since most of the observations were performed on the polycrystalline specimens of commercial purity, which may have a lower stacking fault energy than pure nickel, observations were also



**Figure 2** Another type of the plastic zone formed in polycrystalline nickel. The plastic zone is broad and the dislocations in the plastic zone are perfect dislocations.

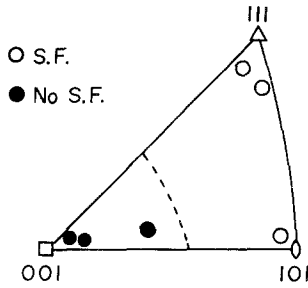


Figure 3 The relationship between the tensile axis orientation and the structure of the plastic zone. The open and the closed circles represent the orientations where the dislocations within the plastic zone are found to be either dissociated or undissociated, respectively.

made on zone-refined single crystal specimens of high purity in order to evaluate the possible effect of specimen purity. A typical example of the plastic zone observed in those specimens is shown in Fig. 4. The dislocation splitting is again observed within the plastic zone, indicating that the observed splitting of the dislocations was not sensitive to the purity of the samples.

#### 4. Discussions

Since the stacking fault energy of nickel is reported to be 120 to  $\sim 150 \text{ mJm}^{-2}$  [5, 6], the observation of partial dislocations and stacking faults is somewhat surprising. In the following, the possible cause for this anomaly will be considered.

It is reported that the stacking fault energy of

pure metals is reduced by alloying [6]. Our observations in pure nickel indicate that the impurities are not the primary cause of the splitting. It is possible that specimen contamination occurs during specimen preparation, especially during electropolishing. Carpenter and Bauer [11] reported that the formation of microtwins was observed after electropolishing thin foils at room temperature. They attributed this to the absorption of hydrogen. There are also several reports suggesting the decrease of stacking fault energy due to hydrogen in nickel [12, 13], copper [14], and stainless steel [15]. The decrease in the stacking fault energy due to hydrogen, however, cannot account for the observed orientation dependence of the structure of the plastic zone.

Copley and Kear [16] pointed out that the resolved shear stresses acting on the leading and trailing Shockley partials are different and the spacing of the pair therefore depends on the orientation of the applied stress. According to their analysis, the steady-state separation  $\Delta\chi$  of a moving Shockley pair is given by

$$\frac{1}{\Delta\chi} = \frac{1}{c} [\gamma \pm 0.5(m_2 - m_1) \sigma b] \quad (1)$$

where  $c$  is a constant which depends on the shear modulus and the orientation of the dislocation line,  $\gamma$  is the stacking fault energy,  $m_1$  and  $m_2$  are the absolute values of the Schmid factor of the leading and trailing Shockley partials, and  $\sigma$  is the

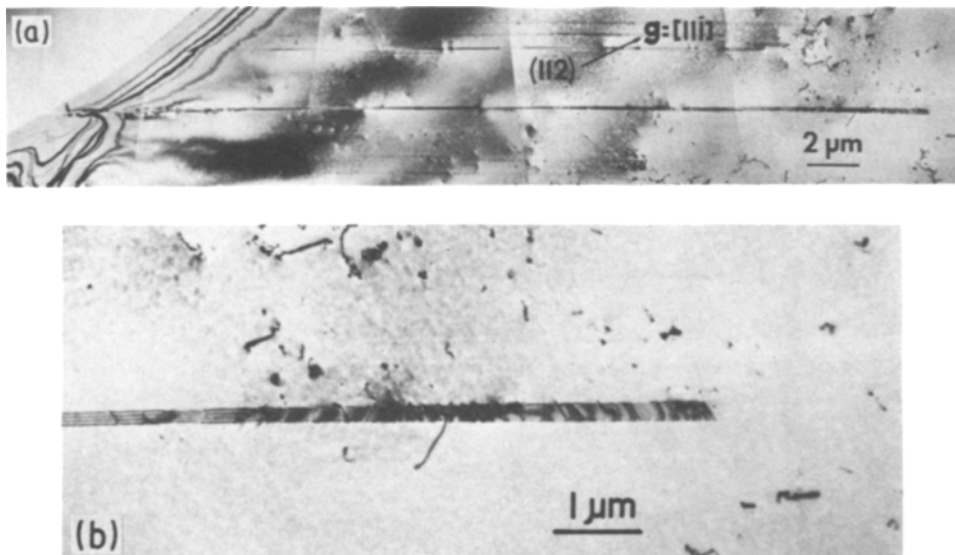


Figure 4 (a) The plastic zone formed in a high purity single crystal specimen. (b) An enlarged view of the end of the plastic zone showing the dislocations which are split into partials.

uniaxial applied stress. For a tensile axis near  $[001]$ , the trailing partial has a higher stress and hence the width of the stacking fault is reduced. On the other hand, for tensile axis near  $[111]$ , the width of the stacking fault is increased since the stress on the leading partial is higher. The boundary between these two regions is along the line connecting  $[102]$  and  $[113]$ . The orientation dependence of the splitting of the dislocations in the plastic zone as shown in Fig. 3 agrees well with this prediction. The magnitude of the effect can be estimated from Equation 1. If  $\sigma = 0$  and  $\gamma = 120 \text{ mJ m}^{-2}$ , the maximum separation is approximately 1.7 nm. Taking the applied stress  $\sigma = 90 \text{ MPa}$ , the maximum separation increases only slightly to 1.8 nm. Thus, the effect of the applied stress on the separation of the partials is too small to explain the observations such as those shown in Figs. 1 and 4.

The orientation dependence of the splitting of dislocations in the plastic zone may be considered in terms of the nucleation of the dislocations from the crack tip. Frank [17] has shown that it is easier to nucleate a partial dislocation than a perfect dislocation provided that  $\gamma$  is not too large. By applying the analysis of Copley and Kear [16], it can be shown that the condition for preferential generation of the leading partial is

$$(m_1 - m_2) \sigma b > 2\gamma \quad (2)$$

This condition is satisfied if  $m_1 > m_2$  and  $\sigma$  is large. For the tensile axis near  $[111]$ , these conditions are satisfied at the crack tip so that the leading partials are generated preferentially. This leads to the pile-up of partial dislocations that are widely separated. On the other hand, for the tensile axis near  $[001]$ , the condition given in Equation 2 is not satisfied so that when the leading partial is nucleated it is even easier to nucleate the trailing partial. Once nucleated the width of the stacking fault is lowered even further because the force on the trailing partial is greater than the leading partial.

Image contrast analysis of the dislocations near the end of the plastic zone in Fig. 1 indicated that most of the partials have the same Burgers vector  $1/6[11\bar{2}]$ . This indicates that all the partials are not necessarily on the same plane, but on the neighbouring parallel planes and the structure observed is possibly a twin lamella similar to that found in copper [2]. Regardless of whether this is, in fact, the case, it is to be expected that the mutual repulsion between the partials on closely

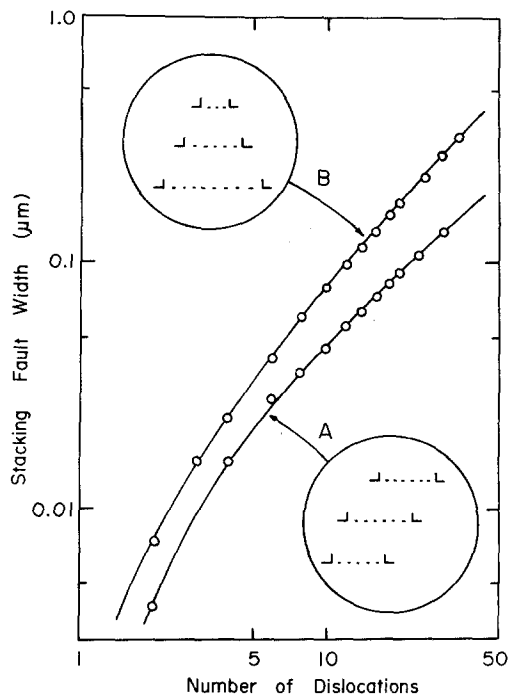


Figure 5 The effect of mutual interaction between the partials on the maximum width of the stacking fault. The insets show the models of the dislocation array assumed for the calculations.

spaced parallel planes as shown in the insets of Fig. 5 may be large enough to separate the partials further. Calculations were carried out to estimate this effect for two simple cases; in the first case (A), each stacking fault overlaps partially and in the second case (B), every pair splits symmetrically (see the insets of Fig. 5). For both cases, the distance between the neighbouring slip planes was taken to be the lattice spacing of the slip plane (0.2 nm). The effect of the distance of the neighbouring slip planes on the width of the separation was found to be relatively small. In Fig. 5, the maximum separation found for each case is plotted as a function of the number of dislocations ( $N$ ) in an array. It can be seen that the configuration B gives rise to a larger separation and the separation of up to  $1 \mu\text{m}$  can be expected for  $N \approx 100$ . This suggests that the separation of the partials can be significantly increased by the mutual interaction of the partials on the neighbouring planes.

From the above considerations, the observed wide separation of the partials and the orientation dependence of the structure of the plastic zone can be explained as follows. The applied stress has

an influence on the nucleation and the initial separation of the partial dislocations. For the tensile axis near  $[001]$ , the applied stress works against dissociation and hence the dislocations remain essentially unsplit. On the other hand, for the tensile axis near  $[111]$ , the nucleation of the leading partial is favoured under the applied stress. The width of the stacking fault is increased further by the mutual interaction of the partials on the neighbouring planes.

## 5. Conclusion

*In situ* electron microscope fracture studies have been performed to investigate the structure of the plastic zone ahead of a crack in nickel foils.

1. Cracks formed in nickel are predominantly shear cracks of mode III type.

2. The structure of the plastic zone is dependent on the orientation of the tensile axis.

3. For the tensile axis near  $[111]$ , the plastic zone is consisted of an inverse pile-up of partial dislocations in the form of a thin ribbon.

4. For the tensile orientation near  $[001]$ , the plastic zone is broader and consists of perfect dislocations.

5. The orientation dependence of the structure of the plastic zone can be accounted for by the stress-induced nucleation and separation of partials.

6. The separation of the partials may be extended by the mutual interaction between the partials.

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