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Probing the Critical Stress Intensity Factor for Slip Transfer across Grain Boundaries by Subgranular Indentation

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ABSTRACT

By analysing the relevant results in the literature, we have found that, when indentation is made on a subgranular level, the hardness varies roughly inversely with the square root of the distance between the indent and the grain boundary. This effect is analogous to the Hall-Petch effect for macroscopic deformation.

INTRODUCTION

It is well-known that the yield strength Y of a polycrystalline material varies with the grain size d according to the Hall-Petch relation

$$Y = Y_o + k_y d^{-1/2} \quad (1)$$

where Y_o and k_y are constants. The Hall-Petch slope k_y is identified as a measure of the ease of slip transmission across grain boundaries. Because of the dependence on the polycrystalline resolution factor and the Taylor factor, k_y is an averaged value of all the grain boundaries within the polycrystalline sample. On the other hand, subgranular microhardness indentation has been routinely carried out in the literature to measure the so-called degree of hardening of grain boundaries [1-4]. In this type of experiments, indentation is often performed near a grain boundary in the edge-on position (Fig. 1), and nearly all the results indicate that the closer the point of indentation to the grain boundary, the higher the measured hardness. In this work, we first conjecture that in the subgranular situation, the hardening effect of the grain boundary is describable by a relation similar to eqn. (1), i.e. the measured hardness H is conjectured to be

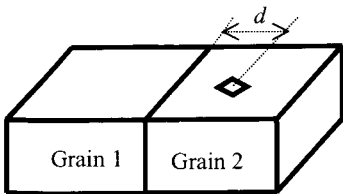


Figure 1. A bicrystal experiment

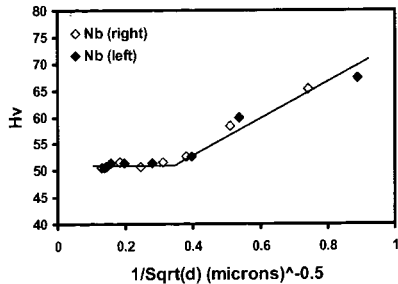


Figure 2. Hardness data for niobium bicrystal. 5 gf load. (data from ref. [3])

$$H = H_0 + k_y' d^{-1/2} \quad (2)$$

where H_0 and k_y' are constants, and d is now the distance of the indent from the grain boundary as shown in Fig. 1. We will check the validity of eqn. (2) using representative results from the literature. We will then develop a mechanistic model to present an interpretation to the meaning of the parameter k_y' in eqn. (2).

ANALYSIS OF EXPERIMENTAL RESULTS

We first use the data by Chou et al [2] on a $[011][\bar{1}\bar{1}\bar{1}]$ symmetric tilt bicrystal of niobium. In Fig. 2 are plotted their hardness values vs $1/\sqrt{d}$, where the open and closed diamonds represent results measured on the "right" and "left" of the grain boundary respectively. The original results show no hardening effect of the grain boundary when d is larger than about $10 \mu\text{m}$ on both sides of the grain boundary. Thus in Fig. 2, H remains roughly constant when the abscissa is less than $\sim 0.3 \mu\text{m}^{-0.5}$. When d is smaller than $\sim 10 \mu\text{m}$, H increases roughly linearly with $d^{-1/2}$ in accordance with eqn. (2). From the slope of the ascending portion of Fig. 2, k_y' for this grain boundary is about $0.30 \text{ MPam}^{1/2}$.

Another example of a bicrystal experiment is the study by Lee et al [4] on Ni_3Al with and without boron doping. Fig. 3 shows the H vs $1/\sqrt{d}$ plot of their results. Again, it can be seen that below a certain d value, the trend given by eqn. (2) is observed. The k_y' values worked out from the slopes of the graphs are $1.45\text{-}1.85 \text{ MPam}^{1/2}$ for the boron-free sample, and $0.90\text{-}1.15 \text{ MPam}^{1/2}$ for the boron-doped samples. The ratio of k_y' without to with boron doping is thus as high as 2.1. This agrees well with the ratio of the Hall-Petch slope of 2.3 obtained from macroscopically yielded samples [5].

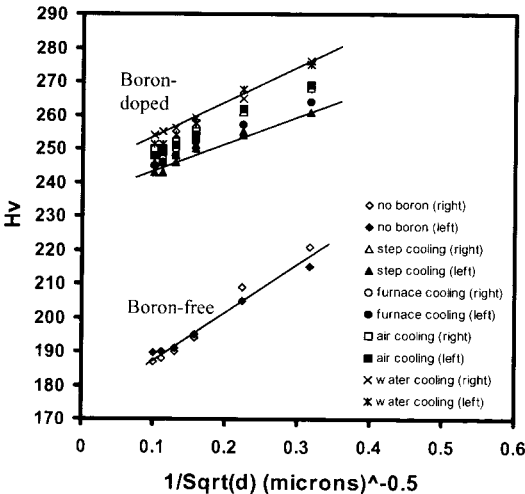


Figure 3. Hardness data for Ni_3Al with and without boron. 10 gf load. (data from ref. [4])

As another type of experiments, we consider the polycrystalline results by Watanabe et al [2] on Fe-1.08 at.% Sn alloy. In such an experiment, microindentation is performed near a grain boundary of a large grain in a polycrystalline specimen. Provided that the distance d of the indent from the grain boundary is much smaller than the grain size, the influence of the remaining curved portion of the grain boundary would be small. Hence the experiment is not dissimilar to one using a bicrystal. In Watanabe et al's experiment, the average grain size was 0.6 mm, and the indent distances over which grain boundary hardening effects were observed were shorter than 100 μm . Fig. 4 shows the H vs $1/\sqrt{d}$ plot of one set of their results exhibiting the least scatter. Once again, an approximate linear dependence in agreement with eqn. (2) is followed. In this case k_y' is about 0.54 $\text{MPam}^{1/2}$.

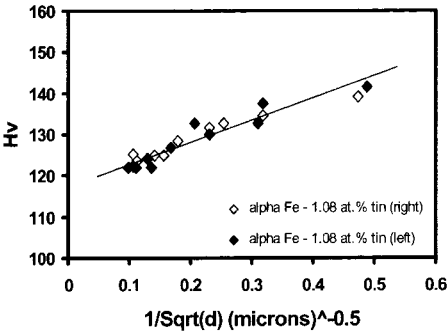


Figure 4. Hardness data for polycrystalline Fe-1.08 at.% Sn. 5 gf load. (data from ref. [2])

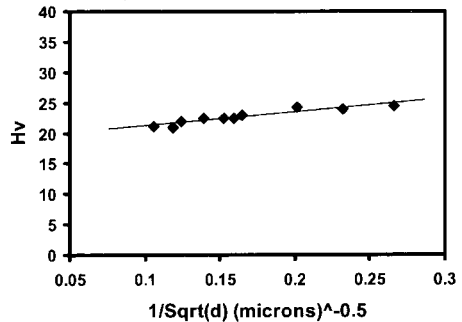


Figure 5. Hardness data for polycrystalline aluminium. 3 gf load.

For further illustration, we have performed a polycrystal experiment using aluminium specimens. The aluminium samples used were cut from a cast ingot (>99.79% pure). The specimens were first homogenised at 450°C for 15 minutes, followed by mechanical polishing down to 1 micron and then electropolishing in 22% perchloric acid and 78% acetic acid at a voltage of 30.5 V. The average grain size was 300 μm . Indentation was then performed on relatively straight grain boundary portions using a Buehler Micromet 2100 microhardness tester with an indentation load of 3 gf. Fig. 5 shows a typical set of the measured hardness plotted against $1/\sqrt{d}$. Again the trend in eqn. (2) is obeyed. From the slope of the best straight line through the data, k_y' is estimated to be about 0.22 $\text{MPam}^{1/2}$.

INTERPRETATION OF k_y'

In Table I, the k_y' values obtained from the present study are compared with the Hall-Petch slopes from the literature. It can be seen that there is a good correlation between the k_y' values and the Hall-Petch slopes. In particular, the k_y' for aluminium is significantly lower than that for iron or Ni_3Al , indicating that, as expected, the strengthening effect of grain boundaries in aluminium is weak. It follows that, like the Hall-Petch slope, k_y' can be used as an indicator for

the ease of slip transmission across grain boundaries. The difference, however, is that k_y' here refers to a selected grain boundary, while the Hall-Petch slope is an average property of the entire polycrystalline specimen.

Table 1. Comparison of k_y' values and Hall-Petch slopes

	k_y' (MPam ^{-1/2}) (present study)	k_y (MPam ^{-1/2}) (Hall-Petch slope)
Ni ₃ Al	1.45-1.85	1.74 [5]
Ni ₃ Al(B)	0.90-1.15	0.78 [5]
Fe	0.54	0.583 [6]
Al	0.22	0.068 [7]

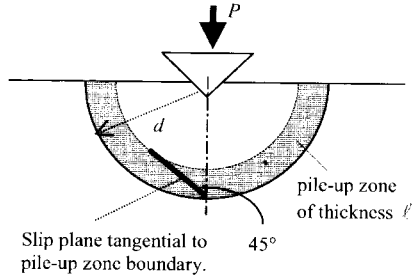


Figure 6. Subgranular indentation.

A simple model for subgranular indentation based on the cavity concept of Johnson [8] may shed some light into the physical meaning of k_y' . Consider for simplicity the situation of making indentation at the centre of a hemispherical grain of radius d as shown in Fig. 6. The picture being considered here is evidently different from that depicted in Fig. 1, but the assumption of spherical field greatly simplifies the treatment here. A concentric plastic zone of radius c is assumed to occur underneath the indent and will spread as the load P increases. As long as the plastic zone sweeps past the grain boundary, dislocation pile-ups at the grain boundary are assumed to result in a pile-up zone of thickness l on the inner side of the grain boundary as shown in Fig. 6. Within the spirit of the cavity model, the region beyond the pile-up zone $r \geq c$ (r = radial distance measured from the centre of the indented grain) is assumed to be purely elastic. The regions $d \leq r \leq c$ and $a \leq r \leq d - l$, where a is the radius of the indent core, are assumed to obey the simple yield criterion $\sigma_\theta - \sigma_r = Y$.

The stress and displacement fields in the elastic region are

$$\sigma_r = \frac{-2Yc^3}{3r^3}, \quad \sigma_\theta = \frac{Yc^3}{3r^3}, \quad u = \frac{Yc^3(1+\nu)}{3Er^2}, \quad r \geq c \quad (3)$$

where E is the Young's modulus and ν Poisson's ratio. In the simple yield region $d \leq r \leq c$, consideration of equilibrium and the yield criterion leads to the following stress solution

$$\sigma_r = -2Y \ln\left(\frac{c}{r}\right) - \frac{2Y}{3}, \quad d \leq r \leq c, \quad (4)$$

which matches the elastic solution in eqn. (3) at the interface $r = c$. In the pile-up region $d - l \leq r \leq d$, assume that as long as the maximum shear stress τ reaches the value given by

$$\left(\tau - \frac{Y}{2}\right)\sqrt{l'} = K_y, \quad d - l \leq r \leq d \quad (5)$$

where ℓ' is an average pile-up length and K_y a critical stress intensity factor, there will be an effective stress relief mechanism operating at the grain boundary so that i) yield will sustain on the other side of the grain boundary in the simple yield region, and ii) τ in the pile-up region will not rise further. In this equation, $Y/2$ represents the lattice friction stress, and K_y is a grain boundary constant having similar nature as k_y in eqn. (1). For example, in the source model, K_y may be taken as $K_y = m' \tau_c \sqrt{r_c}$ where m' is the resolution factor across the grain boundary, τ_c a critical stress to operate a dislocation source on the other side of the grain boundary, and r_c the distance of the source from the grain boundary. The average pile-up length $\ell' \approx d/\sqrt{2}$, in view of the likely situation that a typical slip plane runs at $\sim 45^\circ$ to the grain boundary as shown in Fig. 6. From the equilibrium condition $\partial\sigma_r/\partial r = 4\tau/r$ and eqn. (5), the stress field in the pile-up zone satisfies $2^{1/4} K_y / \sqrt{d} + Y/2 = (r/4)(\partial\sigma_r/\partial r)$. The solution that matches eqn. (4) at $r = d$ is

$$\sigma_r = \left(\frac{4.76K_y}{\sqrt{d}} + 2Y \right) \ln\left(\frac{r}{d}\right) - 2Y \ln\left(\frac{c}{d}\right) - \frac{2Y}{3}, \quad d - \ell \leq r \leq d. \quad (6)$$

For the simple yield region $a \leq r \leq d - \ell$, the stress solution that matches eqn. (6) at $r = d - \ell$ is

$$\sigma_r = 2Y \ln\left(\frac{r}{d - \ell}\right) + \left(\frac{4.76K_y}{\sqrt{d}} + 2Y \right) \ln\left(\frac{d - \ell}{d}\right) - 2Y \ln\left(\frac{c}{d}\right) - \frac{2Y}{3}, \quad a \leq r \leq d - \ell. \quad (7)$$

Consideration of material compressibility [9] leads to the following governing equations for the displacement rate defined as $v = du(r)/dc$:

$$\frac{\partial v}{\partial r} + \frac{2v}{r} = 6(1 - 2\nu) \frac{Y}{E} \left(\frac{v}{r} - \frac{1}{c} \right), \quad d \leq r \leq c \text{ or } a \leq r \leq d - \ell; \quad (8)$$

$$\frac{\partial v}{\partial r} + \frac{2v}{r} = 6(1 - 2\nu) \frac{Y}{E} \left[\frac{v}{r} \left(1 + \frac{2.38K_y}{Y\sqrt{d}} \right) - \frac{1}{c} \right], \quad d - \ell \leq r \leq d. \quad (9)$$

From the above mentioned physical interpretation of K_y , the $K_y/Y\sqrt{d}$ term in eqns. (8) and (9) should not be larger than 1, and since Y/E is very small compared with 1, the v/r terms on the right sides of these two equations are negligible compared with the $2v/r$ on the left sides. An accurate enough solution for both equations is thus $v = A/r^2 - 2(1 - 2\nu)Yr/Ec$, in which the integration constant A has to be the same for all three regions since v is continuous at the interfaces between the regions. By matching this solution to the elastic displacement given by eqn. (3) at the $r = c$ interface, A is found to be $3(1 - \nu)Yc^2/E$, so that the solution for v is

$$v = \frac{3(1 - \nu)Yc^2}{Er^2} - \frac{2(1 - 2\nu)Yr}{Ec}, \quad a \leq r \leq c. \quad (10)$$

Conservation of volume in the indent core implies $2\pi a^2 du(a) = \pi a^2 \tan \beta da$, where β is the angle made by the conical indenter face and the specimen surface. Hence, from eqn. (10),

$$V|_{r=a} = \frac{du(a)}{dc} = \frac{\tan \beta}{2} \frac{da}{dc} = \frac{3(1-\nu)Yc^2}{Ea^2} - \frac{2(1-2\nu)Ya}{Ec},$$

or

$$\left(\frac{c}{a}\right)^3 = \frac{1}{6(1-\nu)} \frac{E \tan \beta}{Y} + \frac{2(1-2\nu)}{3(1-\nu)} \approx \frac{1}{6(1-\nu)} \frac{E \tan \beta}{Y}. \quad (11)$$

The hardness H is the mean pressure in the indent core $-\sigma_r|_{r=a}$ plus $2Y/3$ [8], and so from eqns. (7) and (11),

$$H = H_o + 1.649 \frac{K_y}{\sqrt{d}}, \quad H_o = 2.026Y + \frac{2Y}{3} \ln \left[\frac{E \tan \beta}{6(1-\nu)Y} \right]. \quad (12)$$

The hardness is therefore expressible into a form as in eqn. (2).

Eqn. (2) should really breakdown as d approaches zero. A conjectural suggestion is that d should at least be larger than $(a + \ell)$. Careful inspection of Figs. 2 to 5 reveals that for very small d values, the experimental data indeed fall below the linear trend given by eqn. (2). On the other extreme, when d is too large, the grain boundary would be too remote to interact with the indentation plastic zone. In the above model, this happens when $d > c$. Such an upper bound can be observed in Figs. 2 and 3 as turning points at which the hardness data stop declining with increasing d according to eqn. (2).

CONCLUSIONS

It is proposed that for subgranular indentation, the grain boundary hardening effect can be represented by a relation in which the hardness varies inversely with the square root of the distance of the indent from the grain boundary. Experimental results from the literature confirm the validity of this relation.

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