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Dislocations produced in magnesium oxide crystals due to contact pressures developed by softer cones

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An experimental investigation shows that dislocations, encompassing a predictable dislocated volume, are produced in MgO single crystals subjected to circular contact pressures due to cones of other solids which may be an order of magnitude softer. The mean contact pressure necessary to produce localized plastic flow, without fracture, is shown to be directly related to the critical resolved shear stress of the MgO. Dislocation etching is used to investigate the influence of cone hardness and normal loading on the nature of the deformed zone in the harder crystal and the results are discussed in terms of the deformation characteristics of the softer cones and the crystal.

1. Introduction

The response of contacting surfaces to mutual indentation and consideration of the deformation of surface asperities, with respect to the modelling of both static and sliding couples, has been the subject of considerable study in the past [e.g. 1–3]. Usually, when surfaces of markedly differing hardness are considered, the harder of the surfaces has been treated as relatively undeformable, unless adhesion becomes predominant, and well-established criteria exist to predict the hardness difference necessary to produce macroscopic damage when a softer surface is scratched by a harder one [4]. However, Brookes and Green [5] have demonstrated that harder surfaces can be plastically deformed during static indentation and sliding by significantly softer materials. Furthermore, Brookes *et al.* [6] have recently shown that the cumulative effects arising from repeated deformation of hard surfaces by softer lubricated sliders can lead to wear of the harder surface due to a sequence of dislocation multiplication; dislocation interaction and work-hardening, and finally ultimate and catastrophic fragmentation.

This paper concentrates on the extent to which MgO crystals may be deformed by contact pressures developed beneath cones of significantly softer metals. This is approached by using a dislocation etchant [7] on single crystal MgO surfaces to reveal the distribution and density of dislocations produced by initially sharp cones of the softer material, which subsequently flatten under load to a degree inversely proportional to their nominal hardness. Deformation of the harder crystal is investigated as a function of cone hardness and normal loading.

2. Experimental method

The MgO single crystal surfaces were prepared by cleaving in air followed by chemical polishing in 88% orthophosphoric acid at 100° C. The rate of material removal from the {0 0 1} crystal surface was approximately 6 μm min^{-1} under these conditions. By restricting the polishing time to 2-min intervals, smooth {0 0 1} surfaces were obtained and surface dislocations accidently introduced during cleavage were removed.

A Lietz Miniload microhardness tester was used for indentation of the {0 0 1} MgO surfaces, using cones made from various polycrystalline metals in place of the conventional diamond indenter. Deformation was introduced by lowering the softer cone, having an initial included angle of 136°, on to the {0 0 1} surface of the MgO at a controlled rate giving a "dwell" time of 15 sec on the specimen surface.

Dislocations were subsequently revealed by etching the MgO in a solution of 4 parts NH_4Cl, 1 part H_2SO_4 and 1 part H_2O for approximately 4 min (see [7]). MgO slips on the {1 1 0} ⟨1 $\bar{1}$ 0⟩ slip system at room temperature. Thus, two of the potentially active {1 1 0} slip planes lie at an angle of 90° to a given {0 0 1} surface and four at 45°. The 90° {1 1 0} planes intersect the {0 0 1} surface along ⟨1 1 0⟩ and the 45° {1 1 0} planes in ⟨1 0 0⟩. These planes are hereafter referred to as $\{1\,1\,0\}_{90}$ and $\{1\,1\,0\}_{45}$, respectively. Keh [8] demonstrated that, when observed on the {0 0 1} planes of MgO, dislocation etch pits aligned in ⟨1 1 0⟩ correspond to edge components of expanding dislocation half loops, whilst pits in ⟨1 0 0⟩ correspond to screw components (see Fig. 1).

The dislocation patterns produced in the bulk of the crystal, beneath the indentations, were studied by using the chemical polish outlined above to remove layers from the crystal surface and then re-etching to reveal dislocations on the newly exposed surfaces. The depth to which dislocations had penetrated from the original surface was measured by using "reference squares" of dislocations previously introduced into

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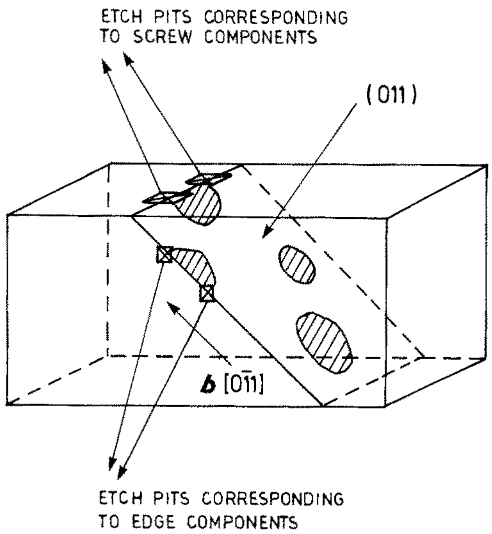

Figure 1 Schematic illustration of the correspondence of etch pits to screw and edge components of expanding dislocation loops, with a [0 $\bar{1}$ 1] Burger's vector **b**, moving on a (0 1 1) plane of MgO.

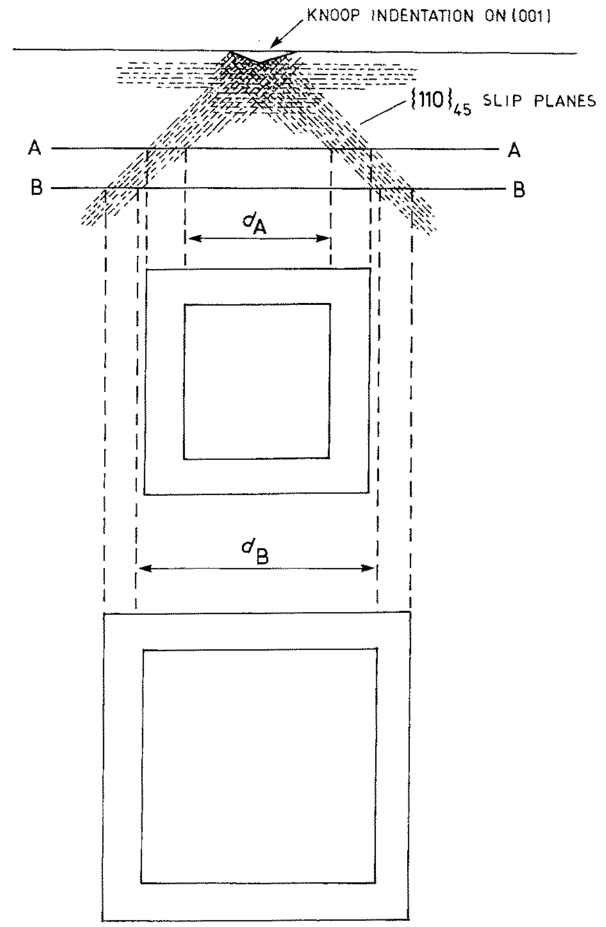

Figure 2 Schematic illustration of depth measurement by the use of "reference squares" of dislocation etch pits. A–A and B–B represent cross sections of the newly exposed surfaces revealed by chemical polishing, d_A and d_B represent the plan view of the dimensions of the square etch pit patterns produced on the new surfaces.

the crystal. Dislocation rosette patterns produced by Knoop indentations revealed a symmetrical square array of etch pits at a range of depths below the original surface proportional to the normal load used for the indentation.

The ⟨1 0 0⟩ edges of the square etch pit pattern were formed by dislocation loops lying on the $\{1\,1\,0\}_{45}$ planes of the crystal. On subsequent re-polishing and etching, the dislocations on these $\{1\,1\,0\}_{45}$ planes formed the perimeter of a larger dislocation-free square, enabling the calculation of the depth of material removed by polishing using the change in dimensions of the square etch pit pattern (see Fig. 2). A number of "reference squares" were introduced into the crystal at various points on the original cleaved surface and measurements were taken each time a layer was removed.

3. Results

3.1. Effect of indenter material on dislocations production

Eight different softer cones of varying hardness were used to investigate the deformation of MgO. Table I lists the materials used with their corresponding hardness values. The hardness of the polytetrafluoroethylene (PTFE) is an approximate figure due to the difficulty in measuring the Knoop indentation produced in this material. In this particular case the indentation was difficult to see and the time-dependent deformation and recovery characteristics of PTFE caused a slow, but perceptible, change in indentation dimensions during measurement.

The hardness of the MgO was determined using Knoop indentations, at a normal load of 100 g (0.98 N) on the {0 0 1}, with the long axis of the indenter aligned with both ⟨1 0 0⟩ and ⟨1 1 0⟩. It can be seen that the hardest of the softer cones used has an indentation hardness well below that of the softest direction in MgO, as the indenter aligned along ⟨1 0 0⟩ and ⟨1 1 0⟩ gave Knoop hardness values of

TABLE I The Knoop hardness of the cones used both before and after loading together with the measured nominal mean pressures developed between the blunted cone tip and the MgO (0 0 1) surface

Cone material	Initial Knoop hardness (GPa)*	Knoop hardness of blunted tip (GPa)*	Nominal mean pressure (GPa)†
PTFE	0.03	0.03	0.02
Lead	0.05	0.05	0.02
Cadmium	0.19	0.19	0.17
Zinc	0.41	0.41	0.29
Aluminium	0.90	1.27	0.80
Copper	0.98	1.37	0.80
70/30 Brass	1.62	2.06	1.27
EN 33 Steel	2.25	3.33	1.71

*Normal load 100 g (0.98 N)
†Normal load 500 g (4.9 N)

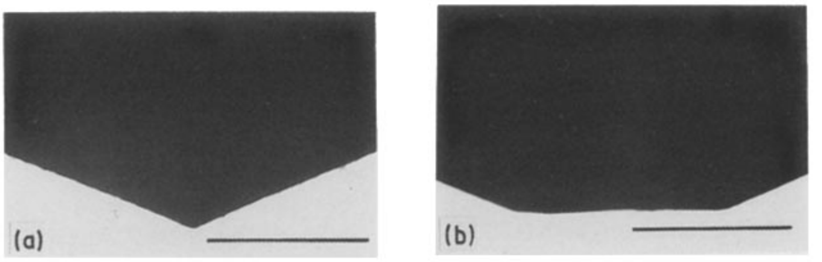

Figure 3 Cross-sectional profile views of a 136° lead cone (a) before and (b) after indentation on MgO (0 0 1) using a 500 g (4.9 N) normal load. (Scale bar = 400 μm).

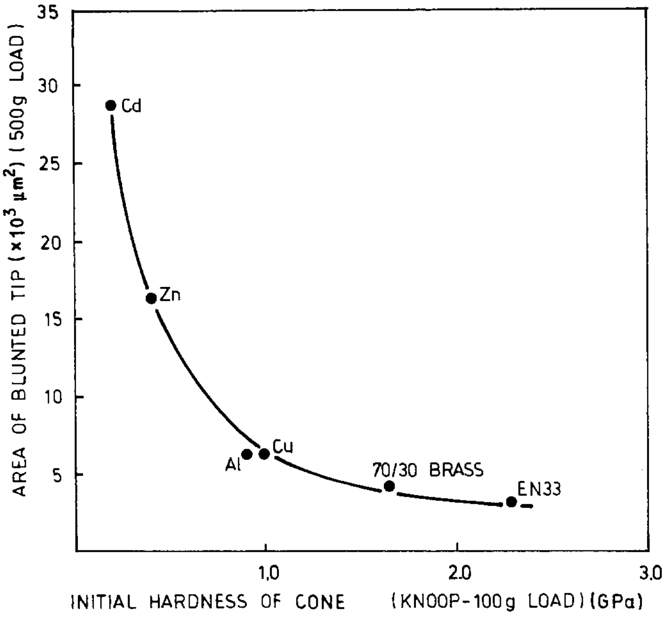

Figure 4 Blunted tip area as a function of cone hardness for the six cones producing dislocations in MgO using a 500 g (4.9 N) normal load.

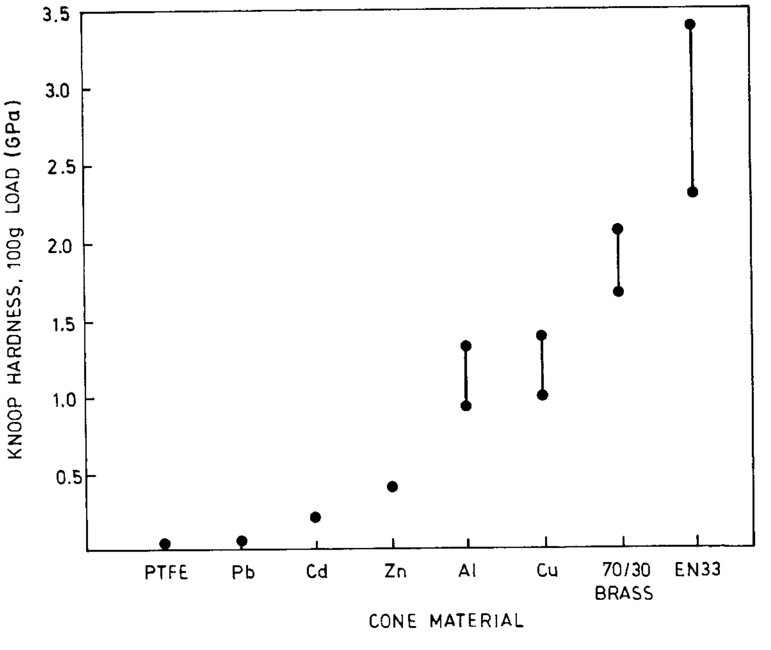

Figure 5 Increase in Knoop hardness of metal cones after a single loading using 500 g (4.9 N) normal load.

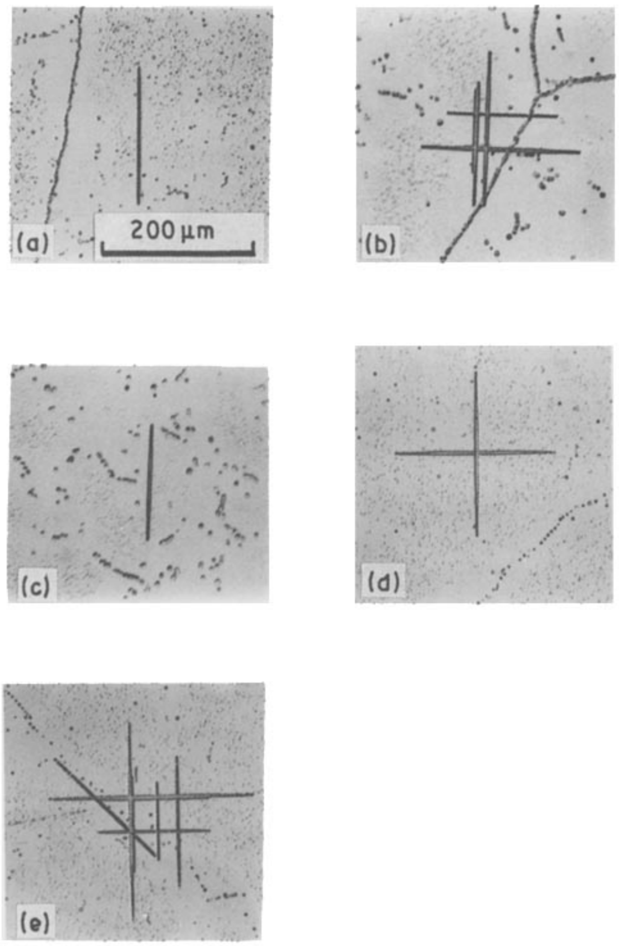

Figure 6 Etch pits produced on (0 0 1) MgO surface after loading using 500 g (4.9 N) normal load. (a) Zinc cone, (b) aluminium cone, (c) copper cone, (d) 70/30 brass cone and (e) EN33 steel cone.

4.70 and 9.65 GPa, respectively. Normal loads of 500 g (4.9 N) were used for the indentation experiments with the softer cones. Predictably, the effect of normal loading on the cones was to cause a blunting of the initially sharp 136° tip. Comparison of a typical cone before and after loading is shown in Fig. 3. The nominal mean pressure developed between the cones and the MgO was determined by dividing the applied load by the measured blunted tip area of the cone. The area of the blunted tip was inversely proportional to the hardness of the cone material as shown in Fig. 4. An increase in the hardness of the cone, associated with the flattening of the tip, was observed in those materials capable of work-hardening at room temperature (see Fig. 5).

The cone materials that showed no change in hardness included PTFE, lead, cadmium and zinc. In the metallic materials, it may be assumed that this was because room temperature is high enough on the homologous temperature scale to ensure that thermal softening effects offset work-hardening processes.

No dislocations were produced in the MgO by the PTFE or the lead cones using applied loads up to 1 kg (9.8 N). The cadmium cone produced dislocations in only one of five indentations, indicating that the critical mean pressure needed for dislocation motion lay in the region of that developed by the cadmium cone. Table I shows the nominal mean pressures developed by the eight metals used at a normal load of 500 g (4.9 N). Dislocations were produced in the MgO crystal consistently by the zinc, aluminium, copper, 70/30 brass and EN33 steel cones. Fig. 6 shows the etch pit patterns produced at the {0 0 1} surface of the harder crystal. It can be seen that the dislocations are confined to a relatively small number of discrete planes with no region of high dislocation density being evident at or, as was subsequently discovered, below the original surface. The EN33 steel cone was the only one to produce dislocations on $\{1\,1\,0\}_{90}$ planes, slip was confined to $\{1\,1\,0\}_{45}$ for all the other cones capable of causing plastic deformation.

The dimensions of the surface etch pit pattern were measured by using the diameter of the smallest circle capable of enclosing all the dislocations revealed at the surface, this will be referred to as the dislocation "winglength". It can be seen from Fig. 7 that the variation in "winglength" dimensions, with hardness

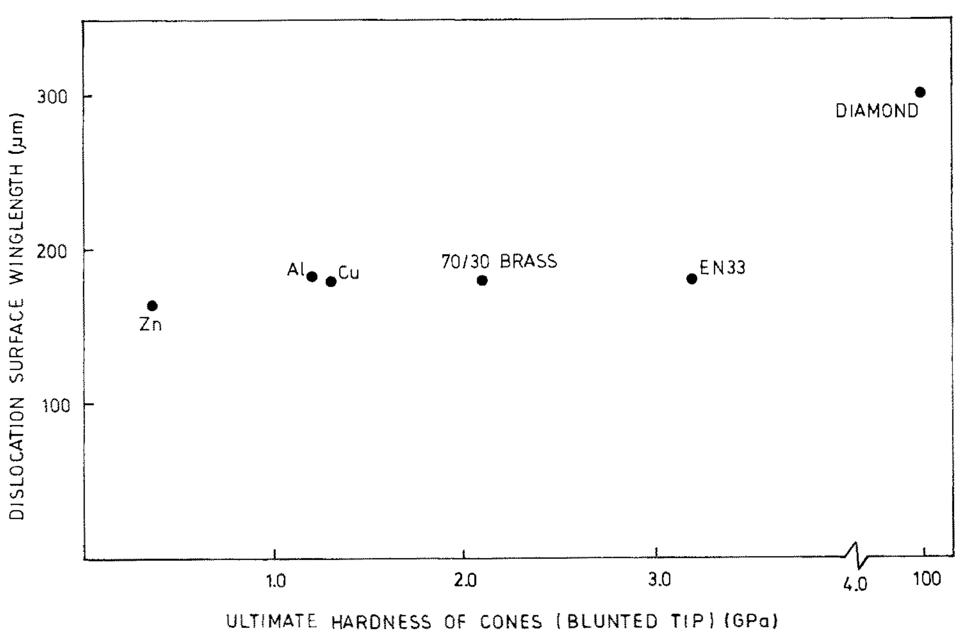

Figure 7 Dislocation surface "winglength" as a function of cone hardness, 500 g (4.9 N) normal load, MgO (0 0 1).

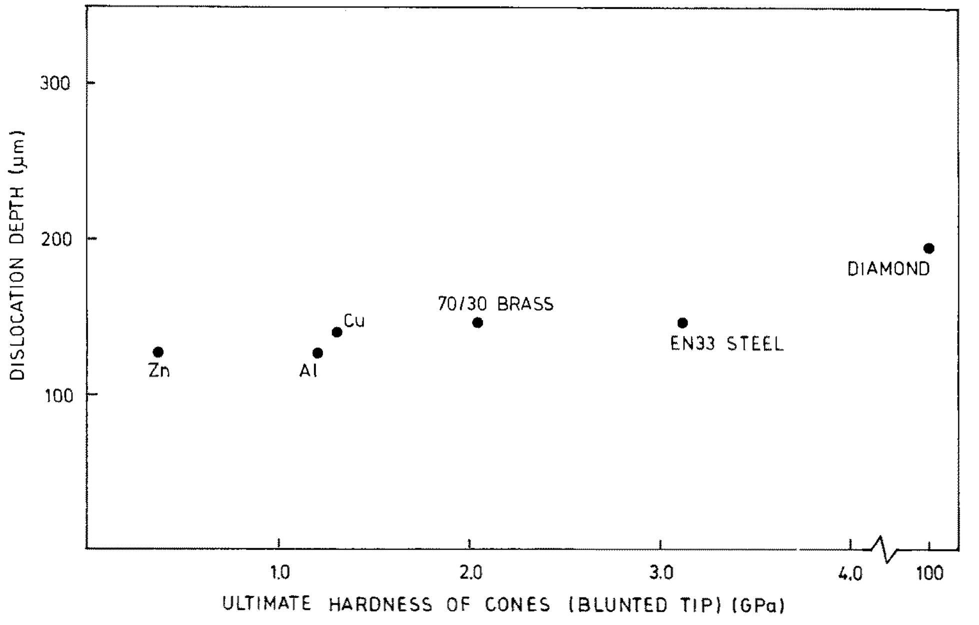

Figure 8 Depth of penetration of the dislocated zone as a function of ultimate cone hardness, 500 g (4.9 N) normal load, MgO (0 0 1).

of the softer metal cone, is relatively small. The dislocations were not confined to the crystal surface but were found to penetrate into the bulk of the crystal. In the case of the EN33 steel cone, the dislocations on $\{1\,1\,0\}_{45}$ were found to penetrate deeper than those produced on $\{1\,1\,0\}_{90}$. The $\{1\,1\,0\}_{90}$ dislocations disappeared at a depth of approximately 50 μm — this compares with a $\{1\,1\,0\}_{90}$ dislocation depth of approximately 80 μm for an indentation produced by a 136° diamond cone. The data shown in Fig. 8 confirms that the depth of dislocations developed beneath the contact area is only slightly dependent on the hardness of the cone.

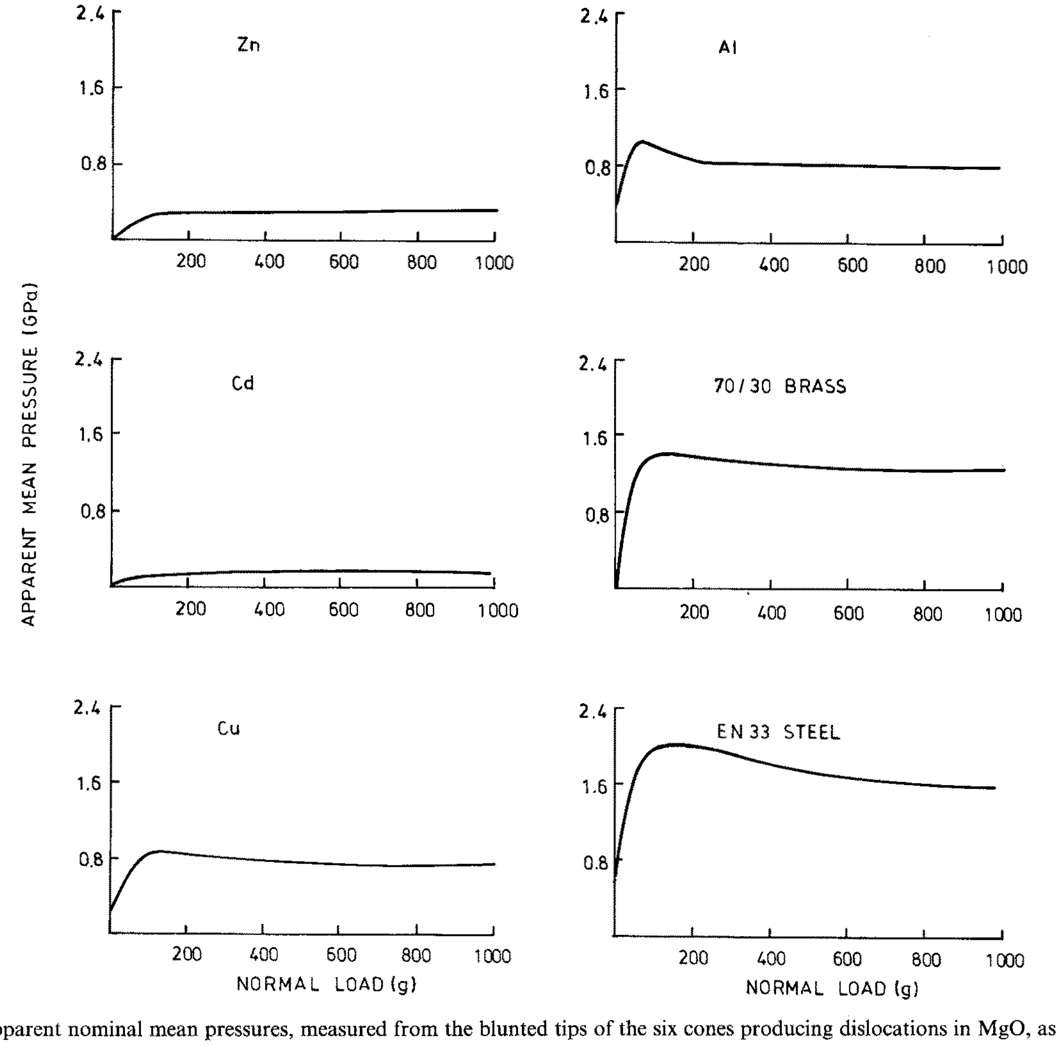

Figure 9 Apparent nominal mean pressures, measured from the blunted tips of the six cones producing dislocations in MgO, as a function of normal load.

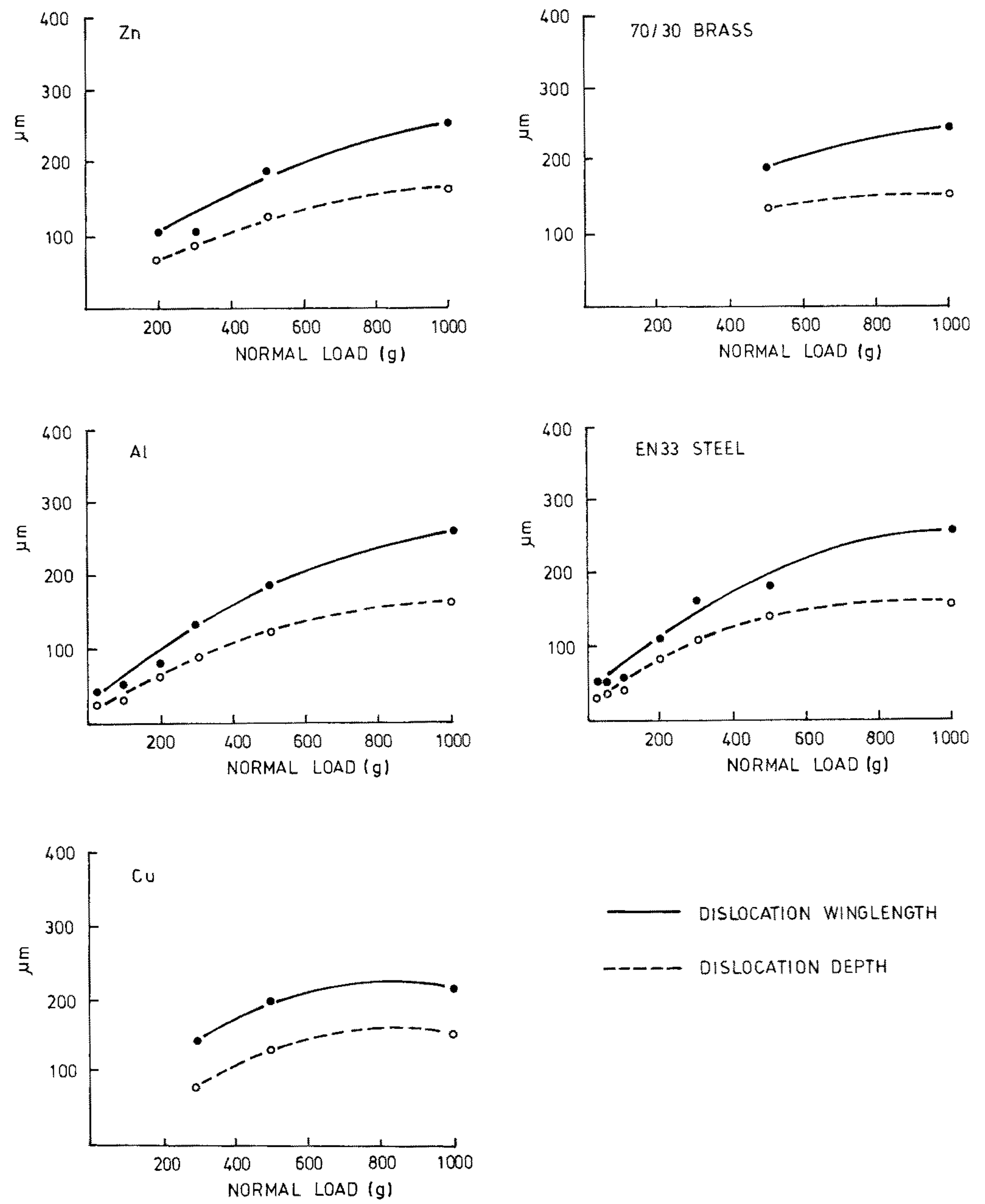

Figure 10 Dislocation surface "winglength" (——) and depth (- - -) of penetration as a function of normal load, MgO (0 0 1).

3.2. Effect of normal loading

Normal loads between 5 g (0.049 N) and 1 kg (9.8 N) were used to investigate the effect of variations in loading on the initiation of dislocation movement in MgO. The results reflecting the effect of normal loading on the nominal mean pressures developed by the cones are summarized in Fig. 9.

A decreasing apparent mean pressure associated with decreasing normal load is clearly evident for loads lower than 100 g (0.98 N) in all cases. Furthermore, there are indications that the maximum nominal mean pressure for each cone tends to occur in the region of 100 g (0.98 N) normal load.

The extent to which dislocation motion is dependent on normal loading is illustrated in Fig. 10, where dislocation depth and surface "winglength" dimensions are plotted against normal load. It is evident that the extent of dislocation motion is generally determined by the normal load, rather than the apparent nominal mean pressure or the hardness of the cone. Fig. 11 illustrates the influence of normal loading on the surface etch pit pattern using the EN33 steel cone.

4. Discussion

The procedure used to calculate nominal mean pressures developed by softer cones was adopted by Mallock [9, 10] as the basis of a particular type of hardness test, termed the Mallock test (see e.g. [11]). Mallock briefly describes a procedure in which small conical specimens of heat-treated steels were slowly loaded against a sapphire plate. Using loads between 0.5 and 5 kg (4.9 – 49.0 N), he found that the Mallock hardness (mean pressure) was virtually independent of load. Further investigations [12–14] using a variety of cone angles and load ranges have given rise to similar conclusions, although some inconsistency has been apparent. More recent work [15] has demonstrated that the cone angles used can influence mean pressures obtained and also that the behaviour of annealed material can differ from work-hardened material in that the mean pressure can decrease with increasing cone angle in work-hardenable materials.

The results of the work described here indicate that for 136° cones at loads between 5 g and 1 kg (0.049 and 9.8 N) the apparent mean pressure (Mallock hardness) developed by a variety of materials is somewhat dependent on normal load – particularly at loads below 100 g (0.98 N). The measured nominal mean pressure decreases at the lower loads. The degree of work-hardening and its influence on the formation of the blunted tip appears to be greater at higher loads, whereas the initially rapid deformation taking place at the cone tips due to relatively high concentrated stresses is reflected by decreased apparent mean pressures.

Inaccuracies in measurement of features approaching the dimensions of the surface finish would be expected to limit the relevance of observations at low loads, however Chaudri and Gilbert [15] have reported similar effects that were additionally influenced by the cone angles used.

When the deformed MgO surface corresponds to a (0 0 1) plane, the resolved shear stress will always be equal on the four $\{1\,1\,0\}_{45}$ planes and will be higher than that on the two $\{1\,1\,0\}_{90}$ planes. The critical threshold contact pressure necessary to produce dislocations in MgO must be directly related to the critical resolved shear stress – which has been observed to lie between 0.049 and 0.069 GPa for deformation in uniaxial compression at room temperature [16]. A recent model, based on the resolution of shear stresses beneath a circular contact with uniform stress, predicts a maximum resolved shear stress of 0.35 *Pm* (where *Pm* = the nominal mean pressure developed by the softer cone) for a (0 0 1) surface of a crystal with $\{1\,1\,0\}$ $\langle 1\,\bar{1}\,0\rangle$ slip systems [17, 18]. This maximum shear stress is situated at approximately 0.33 of the diameter of the contact area beneath the surface and is therefore load dependent for a given cone. Here the softest cone to initiate dislocation movement was cadmium, which developed a mean contact pressure of 0.17 GPa. Assuming this to be the critical threshold pressure for MgO, then the model predicts a critical resolved shear stress of 0.060 GPa which is in good agreement with the value measured using more conventional methods [16].

The symmetry of the dislocated zone did not appear to be significantly affected by the differences in nominal pressure developed by the various metals used for the cones. This is exemplified by the ratios of the surface winglength dimensions to the dislocation depth listed in Table II, for a range of mean pressures developed at a normal load of 4.9 N. This ratio was reasonably constant at approximately 1.3 and can be

TABLE II The ratios of the surface and depth dimensions of the dislocated zones beneath contact areas in MgO (0 0 1) together with the nominal mean pressures developed between the blunted cones and the crystal surface, 500 g (4.9 N) normal load

Cone material	Ratio of surface "winglength"/dislocation depth	Apparent mean pressure (GPa)
Zn	1.3	0.29
Cu	1.3	0.80
Al	1.4	0.80
70/30 brass	1.25	1.27
EN33 steel	1.25	1.71
Diamond	1.5	–

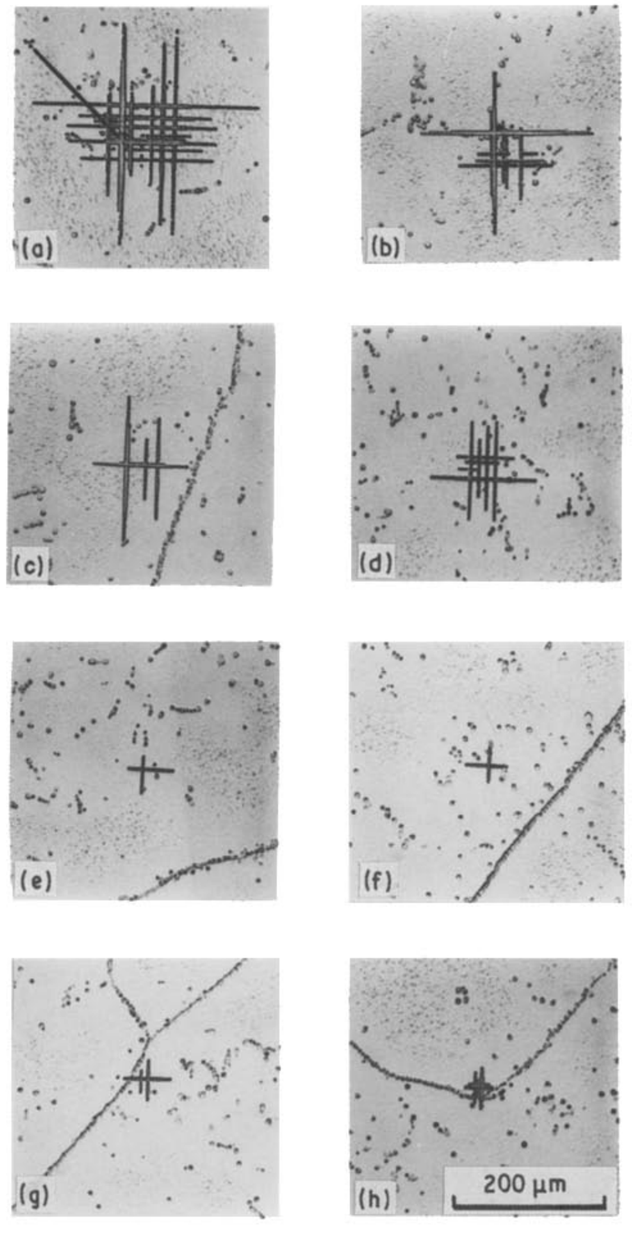

Figure 11 The effect of normal load on the dimensions of the surface etch pit pattern produced on (0 0 1) MgO surface by an EN33 steel cone.

compared with that for a conical diamond indenter, which, in addition to actually penetrating the crystal, gave a ratio of 1.5. However, as the contact pressure increased with the hardness of the cone above the critical threshold level associated with cadmium, the dislocation density on intersecting $\{1\,1\,0\}_{45}$ planes within the deformed zone increased and, presumably, this reflected a greater degree of work-hardening. At still higher contact pressures, i.e. using the EN33 steel cone, dislocation movement on the $\{1\,1\,0\}_{90}$ planes was initiated. This behaviour was consistent with the observations of Swain and Lawn [19] during a study of dislocations produced in LiF by spherical indenters. Using a nylon sphere, they noted that the dislocations were confined to $\{1\,1\,0\}_{45}$ whereas a steel indenter also produced slip on $\{1\,1\,0\}_{90}$. Ultimately, as in the case of a diamond indenter, dislocations are produced on all six $\{1\,1\,0\}$ $\langle 1\,\bar{1}\,0\rangle$ systems and, at room temperature, cracks are formed on the $\{1\,1\,0\}_{90}$ planes by dislocation interactions [20].

5. Summary and conclusions

In summary, it has been shown that considerably softer materials are capable of producing permanent plastic deformation in relatively hard surfaces during static indentation. Given that pressures are developed at the contact area that reach those capable of equalling or exceeding the critical resolved shear stresses on the appropriate slip system, a dislocated volume results, the dimensions of which are primarily determined by the normal load used.

The density of dislocations within this volume increases as the mean pressures developed by the softer cone exceeds that capable of attaining the critical resolved shear stress in the harder crystal. Using a cone angle of 136°, the nominal mean pressures developed by the deforming metal cones are influenced by the normal load, decreasing with loads below 100 g (0.98 N). The measured nominal mean pressures capable of producing deformation in the harder crystal will depend on the specific deformation properties of the softer material, and in particular on the relationship between the real and apparent contact area. However, in the experimental geometry examined here, cadmium, the Knoop hardness of which is an order of magnitude softer than MgO, is on the threshold of being able to initiate dislocation movement in MgO. These results support the application of a simple and novel method for estimating the critical resolved shear stress of ceramic crystals at low homologous temperatures. In addition, they reinforce models and concepts applied to cumulative effects during sliding [6] and suggest that the reality of an undeformable surface during contact of solids may be a rarer event than is commonly supposed.

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