JOURNAL OF MATERIALS SCIENCE 23 (1988) 3321–3325

Plastic deformation of Al_2O_3 single crystals by indentation at temperatures up to 750° C

WOLFGANG KOLLENBERG*
Institut für Mineralogie/RWTH Aachen, 5100 Aachen, West Germany

Vickers hardness measurements have been made on three differently oriented sapphire single crystals in the temperature range from room temperature to 750° C. The hardness generally decreased with increasing temperature but varied with specimen orientation. In the regions surrounding the indents, twinning lamellae and slip lines have been studied systematically and could be associated with dislocation systems. Rhombohedral $\{01\bar{1}2\}$ and basal (0001) twinning as well as prismatic $\{11\bar{2}0\}$ and basal (0001) slip have been observed. Plastic deformation occurred during placement of Vickers microhardness indentations. The hardness was markedly influenced by the number and kind of activated systems. A change in the activated systems caused variation and inversion in hardness ratios. Investigating high-index planes gave much more information on anisotropy effects than would have been obtained had only basally and/or prismatically oriented specimens been used. The latter is commonly the case in the literature. It was shown that in brittle materials, such as ceramics, plastic deformation occurs at temperatures below $0.5T/T_m$ if a stress field with a large hydrostatic component is applied. It is suggested that this is also the case in abrasion.

1. Introduction

Plastic deformation phenomena in aluminium oxide have been studied for more than 30 years. In the available literature it is consistently stated that plastic deformation, due to dislocation activity, is limited to temperatures above 900° C (e.g. [1–3]). Nevertheless twinning has been found to occur at moderate temperatures under compressive loading (e.g. [4]), and Hockey [5] found both, slip and twinning by indentation at room temperature (RT). The successful application of transmission electron microscopy in the study of plastic deformation mechanisms near indents is reported.

Brittle materials may be plastically deformed using a stress field containing a large hydrostatic component. Microhardness measurement involves such a stress field. Therefore the indentation technique is a useful method to investigate the deformation behaviour of brittle materials. The interpretation of Knoop hardness anisotropies is often used to qualify activated slip systems.

The purpose of the present paper is therefore to report the successful application of optical microscopy to study, systematically, slip lines and twinning lamallae near indents and by this means to characterize activated dislocation mechanisms.

2. Experimental procedure

Three specimens (5 mm × 5 mm × 2 mm) with different orientations were cut from a commercial sapphire (Verneuil-grown single crystal). In order to investigate anisotropy effects in orientations other than just the prismatic, two planes at 30 and 60° to the c axis were prepared. The crystallographic orientations (Fig. 1) were confirmed by means of a pole-figure goniometer. The hexagonal indices used throughout this paper are based on the structural rhombohedral unit cell: $c/a = 2.730$ [6]. Specimens were mechanically cleaved and polished. For final polishing various grades of diamond paste were used. The indentation tests were performed at the National Physical Laboratory, Teddington, UK. The hot-hardness tester used has been described elsewhere (e.g. [7]). Specimen and indentor were heated by separate furnaces at the same temperature in a vacuum.

For every specimen the series of indentations started at RT followed by 150° C and then at succesive

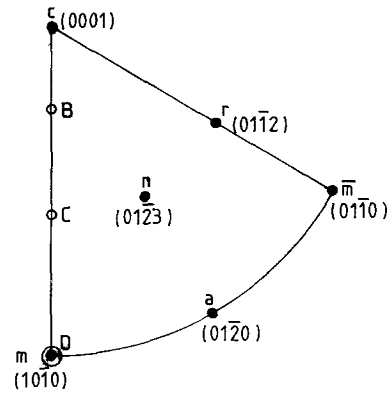

Figure 1 Specimen orientations B, C and D in the stereographic projection of sapphire with regard to the main structural indices and forms.

* *Present address*: PLR, KFA Jülich GmbH, 5170 Jülich, West Germany.

0022–2461/88 $03.00 + .12

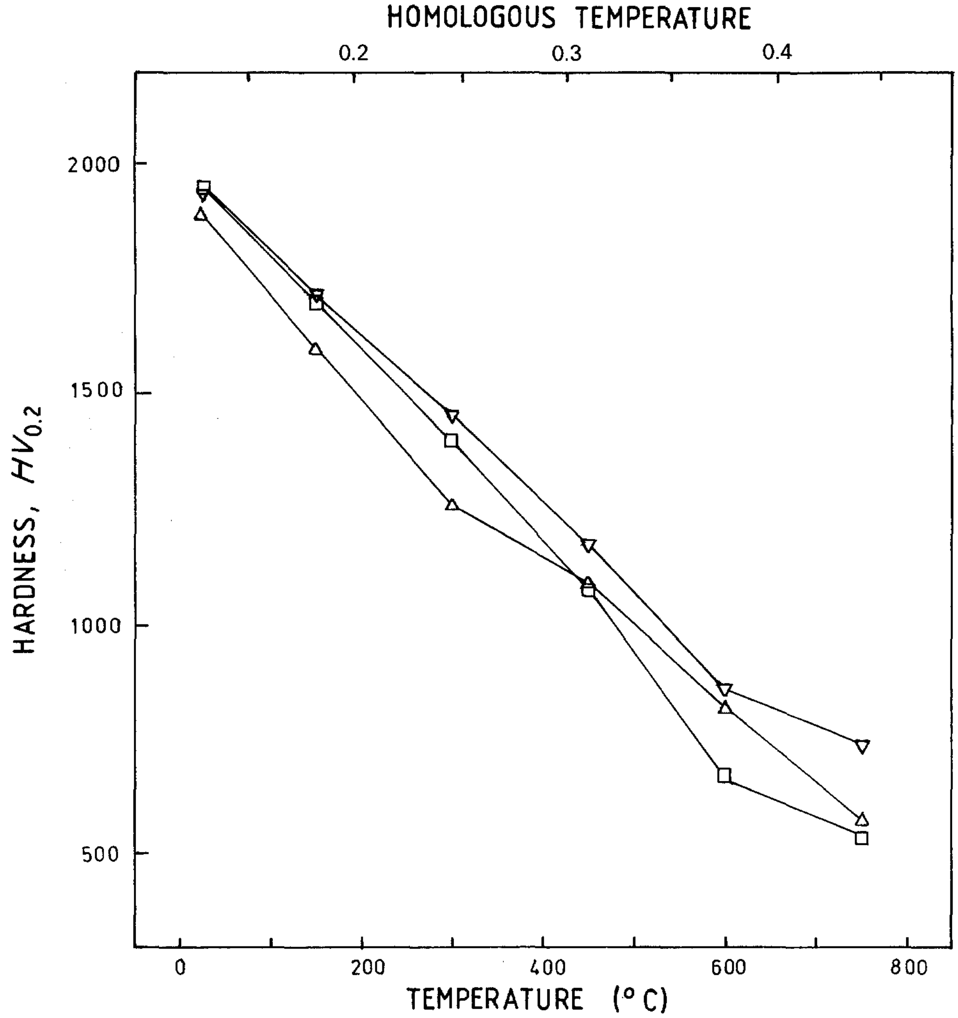

Figure 2 Vickers hardness against temperature for three investigated sapphire specimens of different orientations: (△) B, (▽) C, (□) D.

intervals of 150° C. A Vickers diamond indentor with loads of 1.96 and 3.92 N was lowered for 15 sec at each temperature.

3. Results and discussion

3.1. Hardness

The temperature dependence of hardness for the three differently oriented specimens is shown in Fig. 2. The indentor diagonals were measured after cooling by means of a Leitz Durimet Pol with digital micrometer eyepiece. Each value represents the mean of three indents all having the same alignment.

As expected, the hardness decreased with increasing temperature. Regarding the hardness as related to specimen orientation, an increasing scatter of data at higher temperatures is obvious. This leads to the

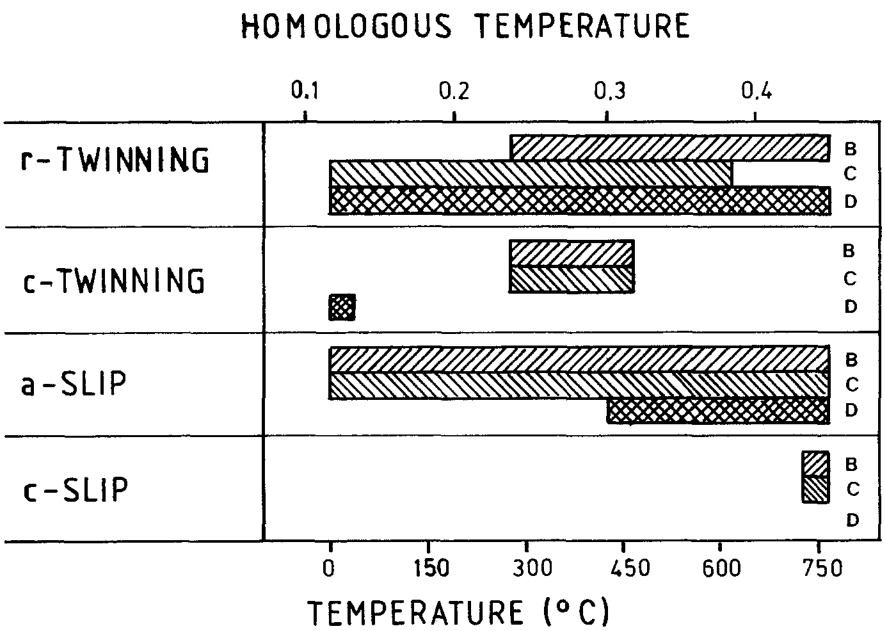

Figure 3 Slip and twinning found to occur between RT and 750° C by microindentation for three investigated sapphire specimens of different orientations.

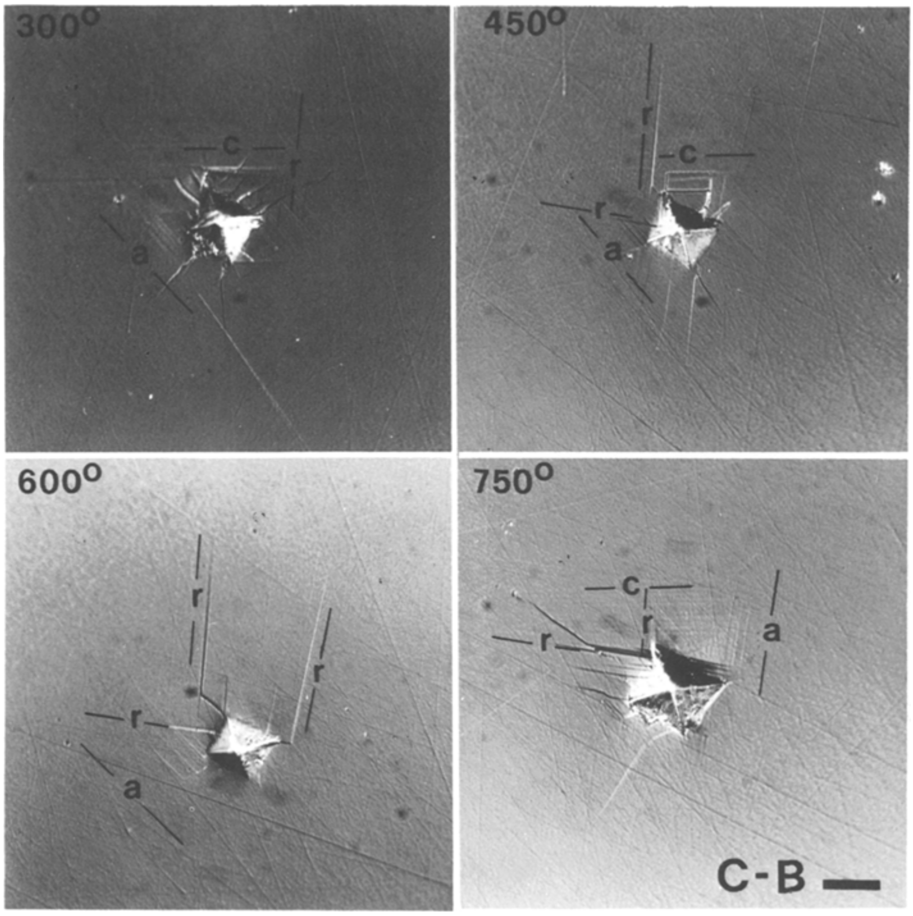

Figure 4 Vickers indentations at 300, 450, 600 and 750° C on Orientation B. Twinning lamellae and slip lines in regions surrounding the indents (bar = 20 μm).

conclusion that the degree of hardness anisotropy rose with increasing temperature. In isostructural haematite a contrary behaviour has been observed [7], where the degree of anisotropy decreased with increasing temperature. This is a common occurrence if no change in the primary slip system is assumed. A reversal in the nature of hardness anisotropy, as observed here, is known for metal carbides [8].

3.2. Slip lines and twinning lamallae

Regions surrounding the indents were examined using optical microscopy and micrographs were taken to define the activated systems. By this method $\{01\bar{1}2\}$ and (0001) twinning and $\{11\bar{2}0\}$ and (0001) slip could be identified. Fig. 3 summarizes the temperature ranges where each system was found to occur in the orientations investigated here. In the following description the letters given in Fig. 1 are used instead of indices to label the planes and systems.

3.2.1. Orientation B

At RT and 150° C only a-slip (see Fig. 4) was found. At 300° C (Fig. 4) both r- and c-twinning were additionally activated. The same was found at 450° C, but at 600° C c-twinning could not be detected and at 750° C c-slip was visible. This could easily be distinguished by the lamella type. The number of r-twin types increased with temperature.

3.2.2. Orientation C

At RT and 150° C r-twinning and a-slip were activated, while c-twinning was additionally found at 300 and 450° C (Fig. 5). At 600° C no c-twinning occurred and at 750° C also r-twinning was not found, but c-slip was then activated. As in orientation B the number of r-twin types increased between 300 and 600° C. As seen in Fig. 3 the behaviour in B and C was very similar; only the temperature range where r-twinning was activated differs.

3.2.3. Orientation D

In this prismatic orientation the plastic behaviour was totally different to that seen in B and C. Firstly at 450° C a-slip was found (Fig 6). The only dislocation mechanism below $0.3T/T_m$ was twinning whereby only c-twinning was activated at RT. In this orientation c-slip was not found to occur.

As presumed above, the increasing degree of anisotropy is caused by changes in the primary slip systems. In Orientation C two systems, r-twinning and a-slip, were activated and possibly impeding each other, a process which could cause the high hardness. Orientations D and B show a second activated system at elevated temperature. The r-twinning regime in D was characterized by higher hardness values than the a-slip regime in B. At approximately $0.3T/T_m$ the hardness ratio was inverted (Fig. 2). The activation of r-twinning in Orientation B at these temperatures led to increasing hardness in B compared to D, where a-slip was additionally activated.

4. Conclusion

By means of microhardness indentation, activated dislocation systems at temperatures below $0.5T/T_m$

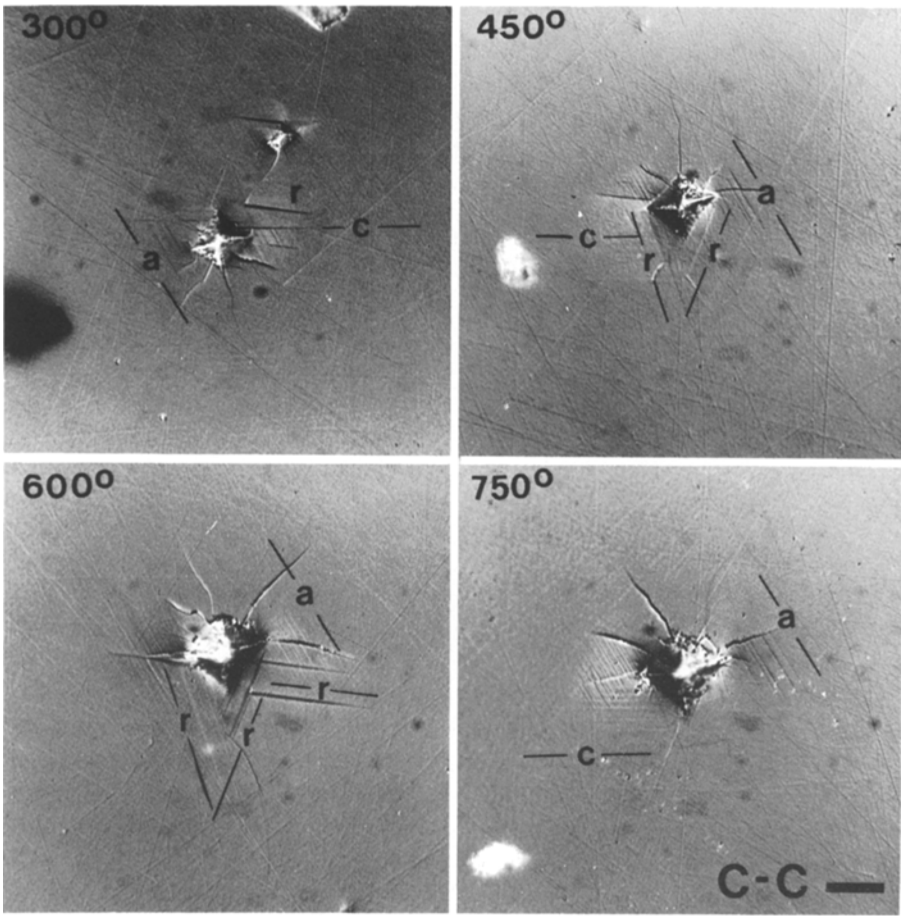

Figure 5 Vickers indentations at 300, 450, 600 and 750° C on Orientation C. Twinning lamellae and slip lines in regions surrounding the indents (bar = 20 μm).

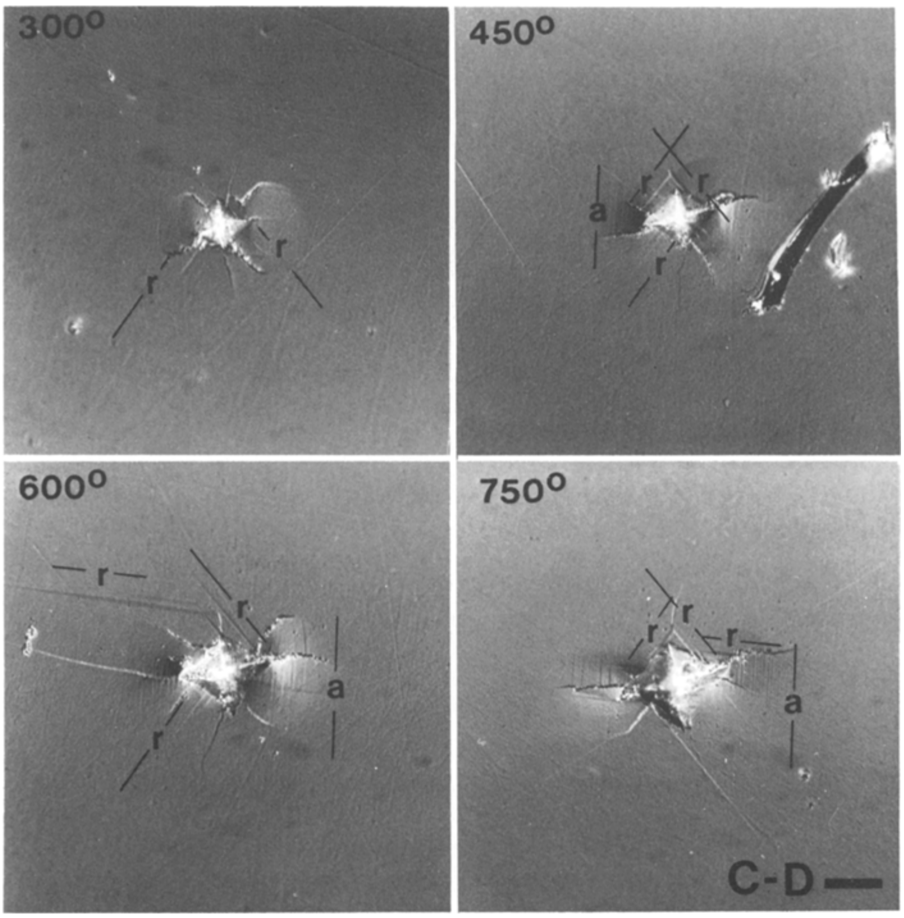

Figure 6 Vickers indentations at 300, 450, 600 and 750° C on Orientation D. Twinning lamellae and slip lines in regions surrounding the indents (bar = 20 μm).

were investigated in sapphire single crystals. Three different orientations, one prismatic and two non-prismatic (30 and 60° to the *c*-axis) were used, and the hardness as well as the activated dislocation systems differed significantly. Although the prismatic plane (Orientation D) showed the highest hardness value at RT, it was found to have the lowest hardness at 750° C. The hardness was markedly influenced by the number and kind of activated systems. From a comparison of B, C and D it becomes evident that it is necessary to investigate not only basal and prismatic planes, as is common in the literature, but especially high-index planes.

The increasing degree of hardness anisotropy is seen as an effect of changes in the primary dislocation systems. Consequently for isostructural haematite no change in primary dislocation systems can be expected when examining the same orientations. This is the object of a current study [9].

It was shown that plastic deformation occurs in Al_2O_3 at low temperatures when applying a stress field with hydrostatic components. It is suggested that microindentation is therefore a powerful method for investigating plastic deformation phenomena even in brittle materials, such as ceramics, at moderate temperatures. Using this method a systematic investigation is possible with only a few specimens and in a short time. As well as under indentors, large pressures are produced locally under irregularly shaped abrasive particles [10]. Therefore, under abrasion the near-surface region may be plastically deformed even at low temperatures. A particularly interesting question, brought out by the present results, is whether an adjusted fabric in near-surface regions may increase the fracture toughness by easing dislocation glide instead of crack propagation. To elucidate the relation between brittle fracture and dislocation generation and motion at low temperatures, additional research will be necessary.

Acknowledgements
E. Almond and D. Clinton are thanked for hospitality and help during my stay at the National Physical Laboratory, Teddington, UK, where I used the hot-hardness tester. Thanks are due to the staff of the Institute for Mineralogy/RWTH Aachen.

References
1. D. M. KOTCHICK, *J. Amer. Ceram. Soc.* **63** (1980) 429.
2. F. W. KLEINLEIN, in "Bruchverhalten keramischer Werkstoffe", edited by D. Munz (Fortschr. -Ber. VDI-Z-18) **11** (1981) p. 32.
3. E. DÖRRE and H. HÜBER, "Alumina" (Springer, Berlin, 1984) p. 135.
4. W. D. SCOTT and K. K. ORR, *J. Amer. Ceram. Soc.* **66** (1983) 27.
5. B. J. HOCKEY *ibid.* **54** (1971) 223.
6. M. L. KRONBERG, *Acta Metall.* **5** (1957) 507.
7. W. KOLLENBERG, *J. Mater. Sci.* **21** (1986) 4310.
8. C. A. BROOKES, in "Science of Hard Materials", edited by R. K. Viswanadham, D. J. Rowcliffe and J. Gurland (Plenum, London, 1983) p. 181.
9. W. KOLLENBERG, *Acta Metall.*, submitted.
10. B. J. HOCKEY, in "Deformation of Ceramic Materials", edited by R. C. Bradt and R. E. Tressler (Plenum, New York, 1974) p. 167.

Received 23 September 1987
and accepted 26 January 1988