

Fractographic Studies of Impact Damage in Single Crystal Sapphire

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충격에 의한 단결정 Sapphire 의 파면 조직에 관한 연구

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초 록

상온에서 단결정 Sapphire가 충격에 의해서 파괴될 때에 수반되는 미세 조직변화에 대하여 관찰하였다.

파괴된 시편을 광학현미경으로 조사한 결과 cleavage는 주로 rhombohedral plane에서 일어나고 있음을 알았다. 그러나 관찰시료 파면의 양상을 박막 (replica)으로 만들어 투과형 전자현미경으로 관찰한 결과로는 국부적으로 소성변화가 일어나고 있음을 알 수 있었다.

이러한 국부적인 소성변화는 crack의 진행을 저해하거나 또는 진로를 변경 시켜주므로 보다 높은 fracture energy를 유발시키는 원인이 될 수 있다.

INTRODUCTION

Oxide ceramics in general do not retain structural integrity under large plastic strains ($\epsilon > 0.01$), and are even less likely to do so under impact conditions. They are nominally brittle unless heated quite high temperatures (typically $> 1000^\circ\text{C}$), and they almost invariably respond to sufficient stresses at all lower temperatures by fracturing. Clearly, these nominally brittle, yet strong materials have an elusive, but significant, quality of impact resistance (specifically, an ability to absorb impart energy), which is somehow associated with their fracture behavior. Because of major discrepancies

between measured fracture surface energies, γ_f , and calculated chemical surface energies, γ_s , it is difficult to understand how a nominally brittle material displaying these significant levels of resistance to impact can be totally brittle in the classical sense of the Griffith crack theory, which equates the release of elastic strain energy only with the chemical surface energy, γ_s , of the new crack surfaces being generated. Because these experimental discrepancies seem too well documented to be lightly dismissed—even in the case of nominally brittle ceramics—one is forced to consider, and investigate experimentally, the possibility of additional energy absorption akin to Rowan's plastic work, γ_p , in the intense stress fields which exist under conditions of

quasi-hydrostatic restraint and near the tips of propagating cracks during impact events.

This paper is aimed to study fracture surface of impact damaged sapphire single crystals by electron fractography to investigate microplasticity and of its role in fracture processes.

MATERIALS

A ballistically damaged specimen of sapphire single crystal was obtained from other investigators concerned with impact resistance of potential light weight armor-materials. The sapphire single crystal was initially 3'' dia \times 3/8 in. thick and had been struck by a 0.36 caliber AP (armor piercing) projectile at a velocity sufficient to cause extensive damage and projectile penetration. The specimen had been cemented to, and was supported by, fiber-reinforced backing material for the test shot.

Fractured fragments were reassembled into the original configuration as well as possible. Macroscopic views of this damaged specimen are shown in Fig. 1. Though they would have been of considerable interest, fine fragments from the immediate impact area were not available for this study.

PROCEDURES

The areas selected for study from the fracture surface were replicated with plastic replicating tape*. The plastic replica was shadowed an evaporated thin film of platinum ($>100\text{\AA}$) impinging at 30° and a thin film of amorphous carbon was deposited at an incident angle of 90° without intervening atmospheric exposure. The plastic first replicas were removed in acetone solution; thereafter, shadowed carbon second replicas were transferred to copper grids for fractography analysis. Details of replication procedures have been given elsewhere¹.

RESULTS AND DISCUSSION

The crystallographic orientation of the sapphire target is presented in Fig. 2 in the form of a stereographic

projection. This pole figure represents the specimen geometric axis and is coincident with the projectile trajectory responsible for the damage shown in Fig. 1 (and approximately parallel with the crystal growth axis). Identities of poles were determined from Laue back reflection X-ray photograms, and were indexed by means of PIMAX tables for $\alpha\text{-Al}_2\text{O}_3$ ^{2,3}.

The planar polished surfaces of the undamaged crystal specimen were nearly parallel with (20 $\bar{2}$ 5) crystallographic planes. The crystallographic orientation of the specimen impact axis can be represented in terms of (1) an inclination of $\sim 30^\circ$ from the [0001] c-axis toward the [2 $\bar{1}$ 10] pole, followed by (2) a counterclockwise rotation of $\sim 13^\circ$ from the [0001]-[2 $\bar{1}$ 10] zone. Alternatively, the axis of impact can be indexed directly as the intercept of two spherical angles, (1) 31.5° from [0001], and (2) 61° from [2 $\bar{1}$ 10].

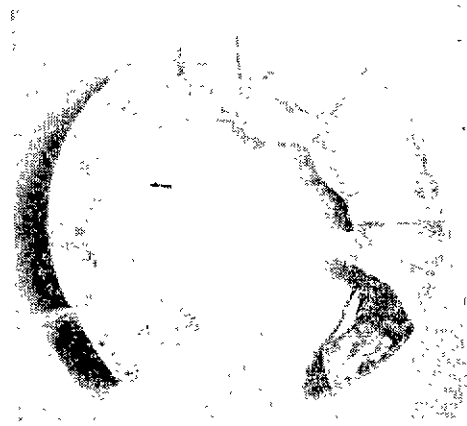


Fig. 1 Overall view of ballistically damaged sapphire single crystal after reassembling fragments, XI.65 (arrow indicate the fragment used for study)

Optical Evidences of Fracture by Rhombohedral Cleavage

The damaged crystal segment (marked) shown in Fig. 1 was indexed by visual inspection in terms of the stereographic projection given in Fig. 2. From this examination, it was found that all the significant crystallographic surface detail could be attributed to cleavage occurring on two first order and two second order rhombohedral planes of the type {101}, {1102}, respectively. All surfaces not so cleaved to have resulted from

*No. 1134 Replicating Tape, a product of Ernest F. Fullam, Inc., Schenectady, N. Y.

conchoidal fracture. The first order rhombohedral cleavage surfaces tended to be planar and smooth, while those of second order were finely stepped or striated. Some of the most prominent crystallographic details are identified in Fig. 3.

Fracture surfaces lying approximately normal to the impact trajectory show a unique sense of directionality as a consequence of the large number of edge intersections of $(10\bar{1}1)$ and $(\bar{1}\bar{1}02)$ cleavage planes. These edges are aligned with the $(\bar{1}\bar{1}01)$ direction, and have characteristic included angles of 133° . Included angles existing between the four operative cleavage systems are indicated in Table 1. Excellent correspondence of predicted and observed angular relationships between specific cleavage planes was observed from somewhat higher magnification micrograph of enclosed area in Fig. 3.

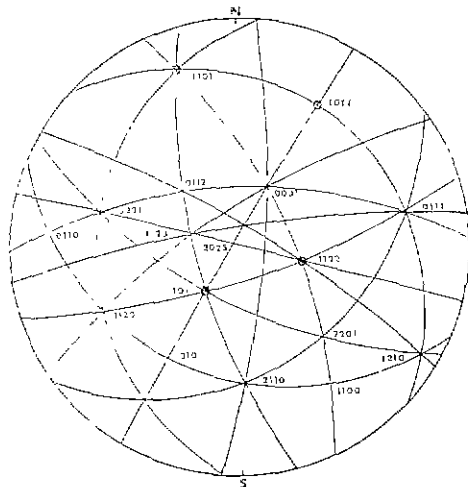


Fig. 2 Stereographic projection showing crystallographic orientation for sapphire fragment marked by arrow in Fig. 1. Indices are given in Miller-Bravais notation for the morphological unit cell, $c/a=1.356:1$. Circled poles correspond to principal cleavage planes (Fig. 3)

Table 1. Angular Relationships Between $\alpha\text{-Al}_2\text{O}_3$ Cleavage Planes. Included Angle* Between Indicated Planes

Planes	$(10\bar{1}1)$	$(\bar{1}\bar{1}02)$	$(\bar{1}\bar{1}01)$	$(\bar{1}012)$
$(10\bar{1}1)$	—	133.01°	86.02°	84.16°
$(\bar{1}\bar{1}02)$	133.01°	—	84.16°	115.18°
$(\bar{1}\bar{1}01)$	86.02°	84.16°	—	133.01°
$(\bar{1}012)$	84.16°	115.18°	133.01°	—



Fig. 3 Identity of cleavage faces on ballistically damaged sapphire fragment, X12.5. Arrows indicates of the poles circled on the stereographic projection in Fig. 2.

Although not evident in Fig. 3, optical examination of other fragments with finely textured cleavage surfaces in reflected or transmitted illumination showed considerable evidence of interference fringes adjacent to cleavage steps. These fringes are indicative of cracks extending along original cleavage surfaces for additional distances beneath the steps. In such cases, it is reasonable to conclude that (1) the break causing an externally visible cleavage step occurred as a consequence of a state of stress which included a bending moment, (2) the crack extending beneath the step along the original cleavage plane was arrested within the bulk after the driving force had been decoupled by the aforementioned break, and (3) the true area of new surface generated by such cleavages exceeds the total externally visible surface. Similar cracks under-running cleavage steps in MgO single crystals have been described by Lewis⁴, who indicated that an overlap of cleavage crack segments appears to occur prior to step formation, and suggested that additional elastic energy absorption would be possible in such processes.

Replication Fractography

The same basic fracture textures stemming from cleavages are maintained only on finer scales, at the higher resolutions and magnifications available through electron

*Included angle between planes is $(180^\circ - \theta^\circ)$, where θ is the angle between pole (normals) to the planes as plotted on the stereographic projection. Included angles are taken from Table II, Ref. 3.

microscopic examinations of plastic-carbon two stage replicas. In Fig. 4, the dominant stepped texture results from three different cleavage planes intersecting at almost orthogonal angles, and is consistent with the expected angular relationship (Table 1)*.

In Fig. 5, a transition can be observed from cleavages of several types in the lower portion to a more finely textured, wavy, and apparently sheared region near the top.

Some cleavage steps are rich in crystallographic detail, as in Fig. 6**, and indicate that conditions comparable to those resulting in cleavage crack segments are also involved in causing the breaks between them. In addition to prominent cleavage, very fine, directionally aligned texturing (indicated by arrows) shown on these



Fig. 4 Cleavage fractography in ballistically damaged sapphire. The three dimensional, almost orthogonal intersections of sets of rhombohedral planes (two first order and one second order) form the basic stepped texture. Arrow indicates other typical oblique intersections between first and second order rhombohedral planes.

*Identities of specific planes and directions have not always been retained through all the stages of replicating, transferring, and magnifying involved in electron microscopic fractography. The four sets of rhombohedral planes previously identified certainly dominate the cleavage textures, but other, less favorably oriented planes may have contributed to the fine textures.

**Figs. 6 and 7 were prepared by a direct replication technique^{5,6}.

cleavage faces is of particular interest, since it appears to indicate some fine scale plastic working during propagation of the cleavage crack. Very local plastic processes, by causing some blunting and/or redirection of the crack tip, could well account for discrepancies between Bruce's⁷ calculated surface free energy for $\alpha\text{-Al}_2\text{O}_3$ (~ 1057 ergs/cm²) and Wiederhorn's⁸ measured rhombohedral cleavage fracture energies for sapphire (~ 6000 ergs/cm²).

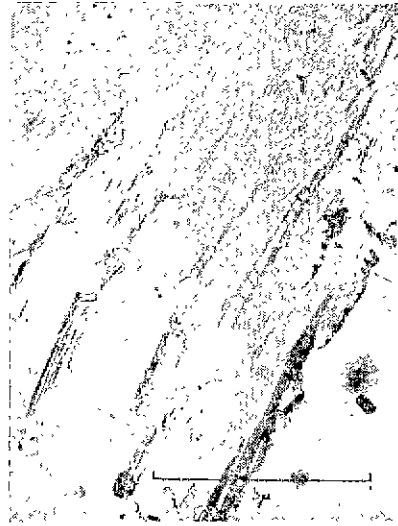


Fig. 5 Transitions from cleaved (lower) to sheared (upper) regions in ballistically damaged sapphire.



Fig. 6 Fine textures associated with cleavage in ballistically damaged sapphire.

Anomalies in an otherwise fairly regular fine cleavage texture are shown in Fig. 7. These anomalies may be associated with interactions between the advancing crack front and slip bands or subgrain boundaries preexisting in the flame grown crystal⁹⁻¹². Alternatively, such textures may have come about as a consequence of stress field perturbations arising from reflections of shock waves.

Although much of the fracture surface observed in ballistically damaged sapphire is distinguished by sharp angularities, and appears to be characteristic of classical cleavage with little evidence of yielding per se, some portions are suggestive of more generalized deformation that, in comparison, is indicative of localized bulk plasticity.

SUMMARY AND CONCLUSIONS

The combined evidences indicate that single crystal sapphire has failed by predominantly cleavage modes during the ballistic impact. Cleavages on a total of four first and second order rhombohedral planes accounted for virtually all the visible and significant crystallographic detail in one extensively studied specimen. At electron microscopic magnifications and resolutions, evidences of permanent deformation processes were observed, but were found only on a very local scale. These limited yielding processes, which appear to some extent to be

capable of blunting or redirecting an advancing crack front, are considered to be consistent with the rather high fracture surface energies ($\gamma_f \sim 6\gamma_s$) reported for rhombohedral cleavage in sapphire by Wiederhorn⁸.

REFERENCES

1. H. Palmour III, C. H. Kim, D. R. Johnson, and C. E. Zimmer (N.C. State University), "Fractographic and thermal analysis of shocked alumina," Technical Report 69-5, THEMIS Research Program on Materials Response Phenomena at High Deformation Rates, Contract N00014-68-A-0187, April, 1969.
2. D. E. Witter and H. Palmour III, pp. 67-101 in Anisotropy on Single Crystal Refractory Compounds, Vol. 1, Edited by F. W. Vahldick and S. A. Merzsol. Plenum Press, New York, 1968.
3. D. E. Witter and H. Palmour III, PIMAK Tables for Alpha Alumina. Engineering School Bulletin No. 84, North Carolina State University at Raleigh, 1967.
4. M. H. Lewis. "Microstructure of magnesium oxide cleavage surfaces," Phil. Mag. 13 (126) 1123-30 (1966).
5. P. L. Gutshall, G. E. Gross, and G. D. Swanson (Midwest Research Inst.), "A study of the physical basis of mechanical properties of ceramics," Air Force Materials Laboratory Technical Report AFML-TR-67-99, Contract AF 33 (615)-2669, June, 1967.
6. P. L. Gutshall and G. G. Shaw; pp. 282-283 in Proceedings of the 25th Annual Meeting of the Electron Microscope Society of America. Sept., 1967.
7. R. H. Bruce, pp. 359-367 in Proceedings of the Second Conference held under auspices of the British Ceramic Society and the Nederlandse Keramische Vereniging at Noordwijk aan Zee, Edited by G. H. Stewart, Academic Press, 1965
8. S. M. Wiederhorn, "Fracture of sapphire," J. Am. Ceram. Soc. 52(9) 485-491 (1969).
9. R. J. Scheuplein and P. Gibbs, "Surface structure in corundum; I. Etching of dislocations." J. Am. Ceram. Soc. 43 (9) 458-72 (1960).
10. J. R. Janowski and H. Conpad, "Dislocations in

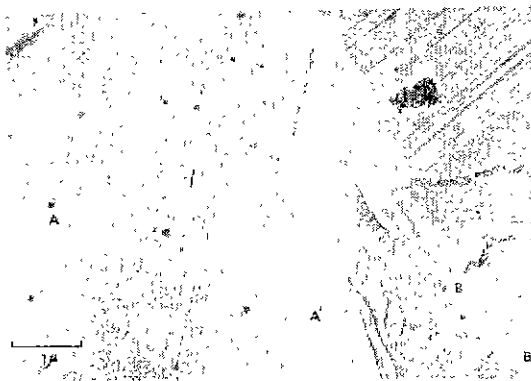


Fig. 7 Abrupt transition from straight to irregular cleavage steps in ballistically damaged sapphire. (Two examples are shown along lines A-A' and B-B')

- ruby laser crystals," *Trans. A. I. M. E.* 230 (4) 717-75 (1964).
11. (a) L. M. Davies, "Residual strain in sapphire rods and boules," *Proc. Brit. Ceram. Soc.* (6) 1-7 (1966).
- (b) L. M. Davies. "The effect of heat treatment on the tensile strength of sapphire," 29-35 *ibid.*
12. J. R. Low, Jr.; pp. 68-90 in *Fracture*, Edited by B. L. Averback, D. K. Felbeck, G. T. Hahn, and D. A. Thomas. John Wiley and Sons, Inc., New York, 1959.