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Vickers Contact Damage of Micro-layered Ti_3SiC_2

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Abstract

The nature, evolution, and degree of deformation-microfracture damage around and beneath Vickers contacts in monophase Ti_3SiC_2 (312) are studied. The 312 material exhibits a pronounced shear deformation during indentation, indicating micro-scale plasticity which can be associated with intra-grain multiple *basal-plane slip between micro-lamellae, intergrain sliding, lamellae or grain push-out, and microfailures at the ends of the constrained shear-slips. No contact-induced cracks are observed and the micro-damage is widely distributed within the shear-compression zone around and below the contacts. The damage process is stochastic which results from a complex interplay of statistical variation in both relative size and crystallographic orientation of individual grains. The ability of 312 to absorb energy from the loading system and to distribute damage is somewhat akin to that of ceramics with either coarse-grained or heterogeneous micro-structures, and perhaps geological structures.*

1 Introduction

Dense polycrystalline samples of pure Ti_3SiC_2 (312) have been recently fabricated by Barsoum and co-workers[1,2] via reactive hot pressing of Ti, graphite and SiC powders at 40 MPa and 1600°C. The 312 material has a hexagonal structure with planar Si layers linked together by TiC octahedra,[3,4] forming a highly deformable basal slip plane.[5] Micro-lamellae of 3–4 μm thick exist within each 312 grain.[1] This material has shown to display a combination of unique mechanical and electrical properties.[1,2] For instance, its electrical and thermal conductivities are higher than those of pure Ti, its thermal shock resistance is comparable to those of metals, and its machinability is similar to that of graphite. With a hardness (Hv) of only 4 GPa and a Young's modulus (E) of 320 GPa, this low ratio of Hv/E suggests that the mechanical behaviour of 312 is somewhat similar to that of ductile metals. The 'ductility' of this material has been verified at room temperature during Vickers indentation and at high temperature (1300°C) during compression tests. In the former, microdamage but no cracks were observed even at the highest indentation load applied (300 N), and the damage was confined to the immediate vicinity of the indentations.[6,7] A number of slip-driven multiple energy-absorbing mechanisms have enabled this material to exhibit micro-scale plasticity near the indentations to inhibit crack formation, and these include:[6] diffuse microcracking, delamination, crack deflection, grain push-out, and grain buckling. During the high temperature compression tests, a pronounced plastic deformation was observed with a yield stress of 250 MPa at 1300°C.[1] These observations suggest that 312 ceramic is damage tolerant, a characteristic which is uncommon for ceramics with a single phase and homogeneous micro-structure. Such property is common only among ceramics with tailor-designed heterogenous micro-structures to impart toughness and crack dispersion.[8–10]

In this paper, results of Vickers contact tests on the 312 material are described. 'Bonded-interface' sections through the Vickers contact zone are used to elucidate the subsurface deformation and micro-mechanisms of damage. The results show that the nature and degree of deformation-microfracture damage in the 312 material is similar to that of ceramics with either coarse-grained or heterogeneous microstructures, and perhaps geological structures.

2 Experimental

Fabrication of Ti_3SiC_2 samples was done using the procedure described elsewhere.[1] Powders of Ti, SiC and C were mixed in the proper molar ratio, cold pressed, and then hot pressed at 1600°C for 4 h at a pressure of 40 MPa. XRD and SEM analysis of sintered samples indicated the presence of single 312 phase with a coarse (100–200 μm) microstructure.[5] Samples for Vickers indentation were ground using SiC grinding paper and polished using diamond paste down to 1 μm.

The microhardness was measured using a Zwick tester at loads (p) in the range 0·2–300 N. At least three measurements were done for each load. Information of subsurface damage during Vickers indentations was obtained using a bonded-interface specimen configuration.[11,12] This test allows the nature and degree of damage accumulation beneath the indenter to be revealed. Polished surfaces of two half-specimens were glued face-to-face with a thin layer of adhesive under moderate clamping pressure. The top surface perpendicular to the bonded interface was polished for the indentation tests. The test to examine the suburface damage was also performed with a Zwick tester at $p=5$, 10, 30, 50 and 100 N. Diagonals of the indenter were centred across and aligned 90° to the interface. The two halves of the indented specimens were then separated by dissolving the glue in acetone, cleaned, gold-coated, and examined using a reflection optical microscope. Details of damaged surfaces at higher magnifications were examined using a scanning electron microscope (Model: Joel 31C).

3 Results and Discussion

3.1 Material characterisation

X-ray diffraction analysis confirmed the presence of 312 as the only phase in the material. Optical microscopy of polished sample under polarised illumination [Fig. 1(a)] shows a wide distribution of grain sizes (10–200 μm) and morphology, and a coarse grain-texture.[6] The anisotropic and layer nature of this material with elongated grains are also revealed in Fig. 1(b). The microstructure with near full densification is evident from SEM examination.

The microhardness of 312 as a function of applied load is shown in Fig. 2. At higher loads, the hardness asymptotes to a value of about1·8 GPa. At lower loads, the hardness values increase with decreasing load levels and reach a maximum value of 3·3 GPa at 2 N. This load-dependent hardness behaviour has also been observed by El-Raghy *et al.*[5] Another feature worth noting is the asymmetry of the indentation which may be ascribed to the anisotropy of material properties.

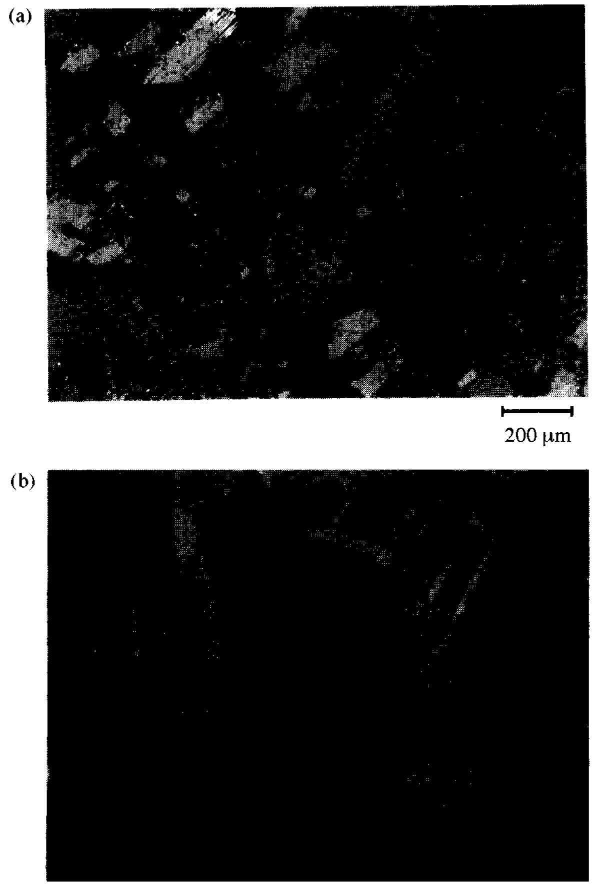

Fig. 1. Micrographs of the 312 material showing (a) grain texture and coarse microstructure in polarised illumination; and (b) fracture surface with micro-layer structure.

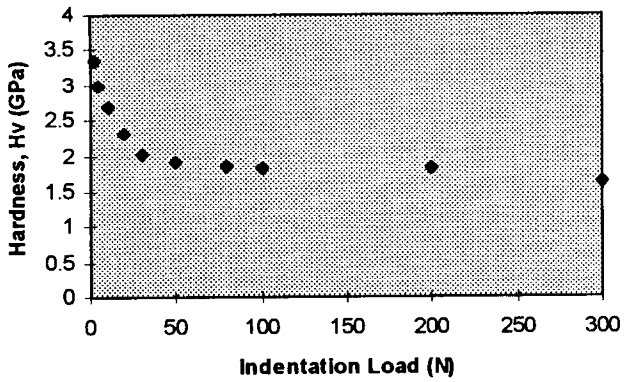

Fig. 2. Variation of microhardness in the 312 material as a function of load.

3.2 Surface and subsurface damage

Figure 3 shows the Vickers indentation damage around the indent at 200 N load. There is a distinct upheaval in the vicinity of the indent as a result of pronounced surface uplift. However, no radial cracks are observed even at the highest load applied, an observation in agreement with others.[5,9]

Optical microscopy confirms that the 'yielding' behaviour during Vickers loading arises from the onset of indentation damage in the 312 material. The nature and evolution of this damage can be discerned from the micrograph sequences in Fig. 4, obtained

Fig. 3. Scanning electron micrograph of the 312 material showing Vickers indentation damage around the indent at 200 N load.

using the bonded-interface section technique previously described. The micrographs show section views of indentations at various loads. The results clearly show the evolution of subsurface damage development in 312 as the load increases. The initiation of the deformation subsurface damage zone, and subsequent expansion of this zone, are immediately apparent from the free-surface relief displacements.

At $p = 5$ N, only a few grains have deformed. At increasing loads, the number of deformed grain increases, and the damage zone expands toward the surface. At $p = 100$ N, the damage is more profuse and begins to take on the appearance of the well-developed, near-hemispherical deformation zone expected from continuum plasticity models.[13] The complete absence of any radial or lateral cracks on the surfaces even at the maximum pressure suggests that 312 is damage tolerant. Radial fracture is inhibited by the ability of the material to contain the extent of microdamage to a small area around the indent via multiple energy-absorbing mechanisms.[6]

The presence of shear-slips traversing the width of some of the deformed grains is clearly evident as deformation-lamellae in the micrographs. A closer examination at higher magnification shows that microcracks extend along those grain boundaries intersected by the lamellae (Fig. 5) due to the concentration of stress intensity. At higher pressures, these microcracks tend to link up with neighbours. However, they appear to be associated only with deformed grains, suggesting that the shear-slips are a necessary precursor to fracture damage in these materials. The variability in orientation of the lamellae is also clearly evident, indicating strong crystallographic features in the damage pattern.

The material appears to undergo 'strain-softening' through shear deformation at increasing contact pressures. This unique behaviour reflects the great propensity of the material to undergo shear-induced deformation at room temperature by virtue of the existence of micro-lamellae within each grain[1] together with an operative basal slip system.[5] This hard metal-like or microscale plasticity at room temperature reinforces the exhibition of substantial gross plasticity at elevated temperatures,[1] and indicates that shear-deformation is either critically stress- or temperature-activated or both. The relatively low value of the Hv/E ratio (0·012) for the 312 material falls in the range 0·001-0·03 which is typical of most metals. This low ratio value has been proposed as an evidence to support its ability to exhibit microscale plasticity.[6]

The contact damage characteristics of Ti_3SiC_2 appears to mirror closely those ceramics with either coarse-grained[11,14,15] or heterogeneous microstructures.[16–19] In particular, the nature of contact-induced damage of 312 is near identical to that of coarse-grained (23 μm) alumina, except for the mode of grain deformation. In both systems, damage is observed to initiate in the subsurface region of high compression-shear beneath the contact instead of in the surface region of weak tension outside the contact. In contrast to coarse-grained alumina, the primary stage of damage in the 312 material involves extensive intragrain basal slip between lamellae, and sliding between grains resulting in surface uplift, grain push-out and grain boundary microcracking. Microcracks are initiated as a result of this slip/sliding process, especially when the pushed-out lamellae impinge at the grain boundary, generating intense stresses. This deformation process is not unlike the pure-shear sliding of geological tectonic plates to cause formation of mountains or earthquakes.[20,21]

3.3 Stochastic shear deformation

The stochastic nature of the deformation-microfracture damage pattern is also evident in Fig. 4. This nature appears to be the result of a complex interplay of statistical variation in both relative size and crystallographic orientation of individual grains. Only those grains of correct size and orientation will favour the occurrence of slip deformation along the basal planes. The expansion of deformation zone is a combination result of the activation of additional grains and/or further basal plane slip systems as the pressure intensifies within the Vickers compression-shear zone. This stochastic phenomenon has also been observed for coarse-grained alumina, although the intrinsic mode is somewhat different.[11]

3.4 Machinability

Another aspect of the microfracture damage process which warrants consideration is the excellent

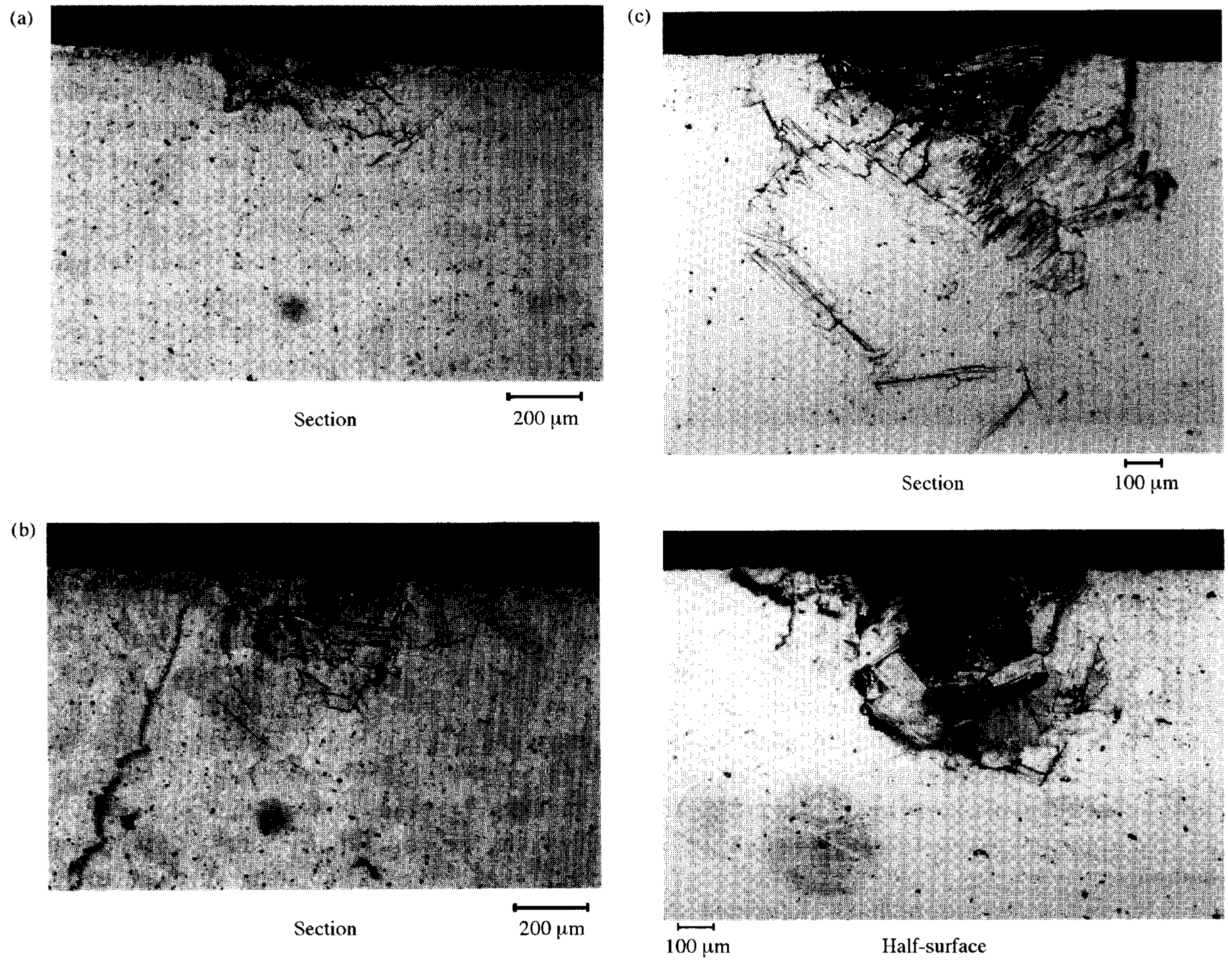

Fig. 4. Optical micrographs showing the section views of Vickers damage in the 312 material. Indentations made with a diamond indenter at increasing contact loads: (a) 10 N; (b) 50 N; (c) 100 N:-section (top); half-surface (bottom).

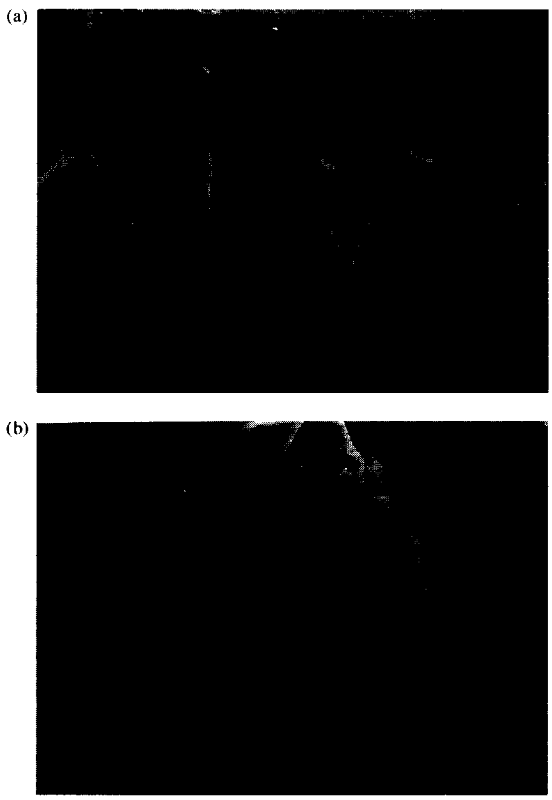

Fig. 5. Scanning electron micrograph showing Vickers-induced (a) shear-slips, and (b) microcracks in the 312 material.

machinability of the 312 material by virtue of its low hardness and micro-layer structure. When compared to conventional oxide ceramics such alumina, the 312 material is approximately 20 times easier to cut in terms of cutting-time using a diamond blade. The key feature of this process is the existence of weak micro-lamellar planes within and between the grains, so that easy shear-slip may occur during machining. Intersection of lamellae with weak grain boundaries allows for a concentration of stress intensity, which in turn facilitates intergranular microcracking. This type of shear fault damage is similar to that observed in coarse-grained homogeneous alumina[11,14,15] and ceramics of heterogeneous microstructures,[22–24] and is responsible for imparting good machinability.[2] The same weak boundaries may also act to suppress macro-fracture by deflecting incipient surface cracks away from the highly directional tensile stress trajectories in their immediate downward propagation. Thus tailor design of ceramic structures with in-situ weak intragrain lamellar planes or intergrain boundaries may offer an effective strategy for improving both machinability and damage tolerance.

4 Conclusion

The capacity of Ti_3SiC_2 for absorbing and distributing damage during Vickers indentations has been demonstrated. Contact damages show characteristics of highly quasi-plastic materials; namely large-scale compression-shear deformation and absence of macrocrack systems. The deformation is accommodated by multiple intragrain slips and intergrain sliding which leads to nucleation of subcritical voids or microcracks. The key to the damage tolerance lies in irreversible deformation and quasi-plasticity under conditions of intense compression-shear stresses beneath the indenter.

Acknowledgements

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References

1. Barsoum, M. and El-Raghy, T., Synthesis and characterisation of a remarkable Ceramic: Ti_3SiC_2. *J. Am. Ceram. Soc.*,, 1996, **79**, 1953–1956.
2. Barsoum, M., Brodkin, D. and El-Raghy, T., Machinable layered ceramics for high temperature applications. *Scr. Mater. Metall.*, in press.
3. Goto, T. and Hirai, T., Chemically vapour deposited Ti_3SiC_2. *Mater. Res. Bull.*,, 1987, **22**, 2295–2302.
4. Arunjatesan, A. and Carim, A. H., Symmetry and crystal Structure of Ti_3SiC_2. *Mater. Lett.*, 1994, **20**, 319–324.
5. El-Raghy, T., Synthesis, structure and properties of Ti_3SiC_2. Ph.D. thesis, Drexel University, Philadelphia, PA. 1996.
6. El-Raghy, T., Zavaliangos, A., Barsoum, M. and Kalidindi, S. R., Damage mechanisms around hardness indentations in Ti_3SiC_2. *J. Am. Ceram. Soc.*, 1997, **80**, 513–516.
7. Lis, J., Miyamoto, Y., Pampuch, R. and Tanihata, K., Ti_3SiC_2-based materials prepared by HIP-SHS techniques. *Mater. Lett.*, 1995, **22**, 163–168.
8. Padture, N. P., In-situ toughened SiC. *J. Am. Ceram. Soc.*, 1994, **77**, 519–523.
9. Lawn, B. R., Padture, N. P., Cai, H. and Guiberteau, F., Making ceramics 'ductile'. *Science*, 1994, **263**, 114–116.
10. Harmer, M. P., Chan, H. M. and Miller, G. A., Unique opportunities for microstructural engineering with duplex and laminar ceramic composites. *J. Am. Ceram. Soc.*, 1992, **75**, 1715–1728.
11. Guiberteau, F., Padture, N. P. and Lawn, B. R., Effect of grain size on Hertzian contact damage in alumina. *J. Am. Ceram. Soc.*, 1994, **77**, 1825–1831.
12. Van Der Zwaag, S., Hagan, J. T. and Field, J. E., Studies of contact damage in polycrystalline zinc sulphide. *J. Mater. Sci.*, 1980, **15**, 2965–2972.
13. Tabor, D., Hardness of Metals. *Clarendon*, Oxford, 1951.
14. Guiberteau, F., Padture, N. P., Cai, H. and Lawn, B. R., Indentation fatigue: a simple cyclic Hertzian test for measuring damage accumulation in polycrystalline ceramics. *Philos. Mag.*, 1993, **A 68**, 1003–1016.
15. Wei, L. and Lawn, B. R., Thermal wave analysis of contact damage in ceramics: Case study on alumina. *J. Mater. Res.*, 1996, **11**, 939–947.
16. Padture, N. P., Pender, D. C., Wuttiphan, S. and Lawn, B. R., In-situ processing of silicon carbide layer Structures. *J. Am. Ceram. Soc.*, 1995, **78**, 3160–3162.
17. An, L., Chan, H. M., Padture, N.P. and Lawn, B. R., Damage-resistant alumina-based layer composites. *J. Mater. Res.*, 1996, **11**, 204–210.
18. Liu, H., Lawn, B. R. and Hsu, S. M., Hertzian contact response of tailored silicon nitride multilayers. *J. Am. Ceram. Soc.*, 1996, **79**, 1009–1014.
19. Pratapa, S., Synthesis and character of functionally-graded aluminium titanate/alumina-zirconia composites. M.Sc. thesis, Department of Applied Physics, Curtin University of Technology, 1997.
20. Allmendinger, R. W. and Gubbels, T., Pure and simple shear plateau uplift, Altiplano-Puna, Argentina and Bolivia. *Tectonophysics*, 1996, **259**, 1–13.
21. Vanderbeek, P., Plank uplift and topography at the central Baikal rift—a test of kinematic models for continental extension. *Tectonics*, 1997, **16**, 122–136.
22. Cai, H., Kalceff, M. A. S. and Lawn, B. R., Deformation and fracture of mica-containing glass-ceramics in Hertzian contacts. *J. Mater. Res.*, 1994, **9**, 762–770.
23. Cai, H., Kalceff, M. A. S., Hooks, B. M., Lawn, B. R. and Chyung, K., Cyclic fatigue of a mica-containing glass-ceramic at Hertzian contacts. *J. Mater. Res.*, 1994, **9**, 2654–2661.
24. Padture, N. P. and Lawn, B. R., Toughness properties of a SiC with in-situ induced heterogeneous grain structure. *J. Am. Ceram. Soc.*, 1994, **77**, 2518–2522.