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The fracture of jade

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Fracture parameters have been measured in jade samples obtained from three sources. Fracture toughness values lie between 2.9 and 5.5 MN $m^{-3/2}$, whereas fracture energies are between 269 and 986 J m^{-2}. The high resistance of jade to crack propagation is related to its fibrous microstructure. In materials from each source, the diameter of individual nephrite fibres was $\sim\frac{1}{2}$ μm, but fibres tend to be grouped in feather-like or cylindrical bundles, with the bundles arranged randomly in a woven type structure. Sub-critical crack growth can occur in jade in the presence of water but not in lapping oil. The fracture behaviour of jade is compared with that of common rocks and engineering ceramics.

1. Introduction

Jade is a collective name for a number of chain silicate minerals which are valued as semi-precious stones. The jade minerals are jadeite, $NaAlSi_2O_6$, which has a simple pyroxene chain structure and nephrite which refers to dense forms of the amphiboles actinolite, $Ca_2Mg_5Si_8O_{22}(OH)_2$, or the iron substituted form tremolite, $Ca_2(MgFe)_5Si_8O_{22}(OH)_2$. Both forms of jade are renowned for their high toughness. This reputation rests on one hand on many archeological finds of jade impacting tools and on the other on the practical experience of gem-cutters. However, no quantitative measurements of the toughness of jade have been reported.† Indeed, such measurements have been made for only a few of the commonest rocks [1–4]. In this case toughness has been evaluated in terms of fracture energy, G, rather than critical stress intensity factor, K_{IC}, which is the more usual determination in the case of metals and ceramics. Typically, G, for rocks lies between about 24 J m^{-2} for a lithographic limestone to about 200 J m^{-2} for granite [2]. For sandstone, a wide variety of values of G has been reported [1–4] which depend upon the source, orientation and possibly, the testing method employed. The strength of rocks is about 100 times less than that of ceramics, but the latter are also not particularly tough, having G values up to 100 J m^{-2}. The toughness of rocks is relevant to mining operations where the object is to fracture the material as easily as possible. In contrast, engineering ceramics should be as tough as possible in order to resist fast moving cracks and thus, retain structural integrity. In the present work, a natural material has been studied which shows some fracture characteristics desirable in engineering ceramics. The main purpose of the work was to determine the toughness of jade and to account for it in terms of the microstructure. A secondary goal was to compare the fracture behaviour of jade with that of ceramics to try and obtain an insight into the levels of toughness which might be achieved in non-metallic materials.

2. Experimental

Jade was obtained from three sources: Alpa di Selva, Poschiavo, Switzerland; Frazer River, British Columbia, Canada; and an unknown location in the Soviet Union. Jades from these sources are all known to be nephrite, which was confirmed by

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†Since this manuscript was prepared, a paper by R. C. BRADT *et al. Amer. Mineral.* 58 (1973) 722, has been brought to the authors' attention. An average value of G for nephrite is reported as 450 J m^{-2}. K_{IC} was calculated as 7.7 MN $m^{-3/2}$ using measured values of G and Young's modulus. The present results indicate that this calculation can over estimate K_{IC} considerably because of the extensive crack branching which occurs during propagation.

X-ray analysis (see below). Fracture toughness, K_{IC}, fracture energy, G, and flexural strength, σ, were all measured in four-point bending. For measurements of K_{IC} and G, the sample dimensions were 50 mm x 5 mm x 2.5 mm for all three materials and for the Selva jade, sample sizes of 150 mm x 15 mm x 7.5 mm and 150 mm x 30 mm x 15 mm were also tested. The flexural strength samples had dimensions 50 mm x 3 mm x 3 mm. All the small samples were lapped with 13 μm SiC on a cast iron wheel. The smallest fracture mechanics samples were notched with an annular diamond saw to give notch dimensions 0.25 mm wide with a tip radius ~0.1 mm. The larger Selva samples were cut and notched with a 200 grit diamond peripheral wheel, and tested without further surface preparation. The notch dimensions were ~1 mm wide with a tip radius ~0.8 mm.

All specimens were air-dried and tested in this condition unless otherwise stated. Tests were performed on an Instron machine at a cross-head speed of 0.05 cm min^{-1} with some preliminary measurements at 0.005 cm min^{-1}. Fracture mechanics tests were carried out according to ASTM procedures for determining the load and assessing the validity of the test [5]. Fracture toughness was evaluated from the relation [6]:

$$K_{IC} = \frac{3Pda^{1/2}\,[3.86-6.15(a/w)+21.7(a/w)^2\,]^{1/2}}{BW^2}$$

where P is the load, d the bending arm, a the crack length and W and B the specimen width and thickness respectively. G values were measured from the area under the load/deflection curve and the total projected area of fracture, i.e. twice the ligament area.

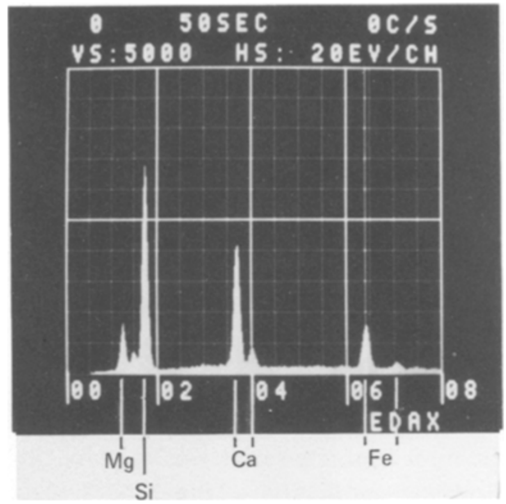

Figure 1 Energy dispersive X-ray analysis for the matrix material in Frazer River jade.

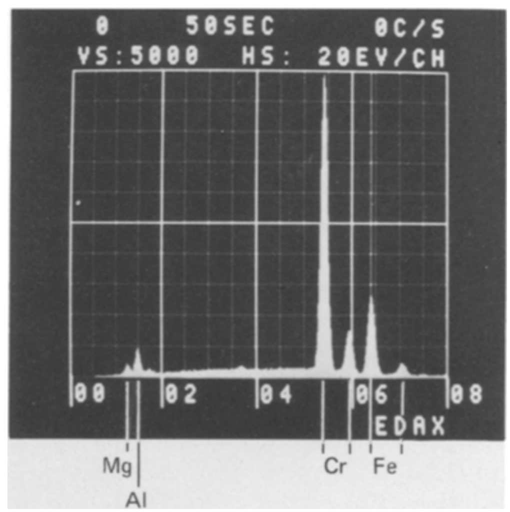

Figure 2 Energy dispersive X-ray analysis of black inclusions in Russian jade.

3. Results and discussion

3.1. Microstructure

Both the Russian and Frazer River jades were translucent stones with similar fine green colours. As is common in green nephrites, both types contained irregular plate-like black inclusions up to $\frac{1}{2}$ mm in size. The Russian jade also contained a few bright green inclusions of somewhat smaller size. Further, a close inspection of the Russian material revealed a number of natural flaws in the form of hairline cracks up to 5 mm long. No equivalent defects were detectable in the jade from Frazer River. The Selva jade was a two-phase material consisting of pale green islands surrounded by white veins but contained no inclusions. The relative sizes of the islands and the depth of their colour varied considerably in a large, roughly cylinderical stone approximately 30 cm x 10 cm. Some regions were essentially free of veins, but generally the islands were about 3 mm and the veins $\frac{1}{2}$ to 1 mm wide. Regions containing obvious flaws were avoided when cutting mechanical test samples from each jade, but the visual detection of small flaws was difficult because of local colour variations and in the case of the Selva jade, because of the presence of the veins.

Energy dispersive X-ray analyses of the matrices of jade from each source were practically identical and showed the presence of Mg, Si, Ca and Fe, as

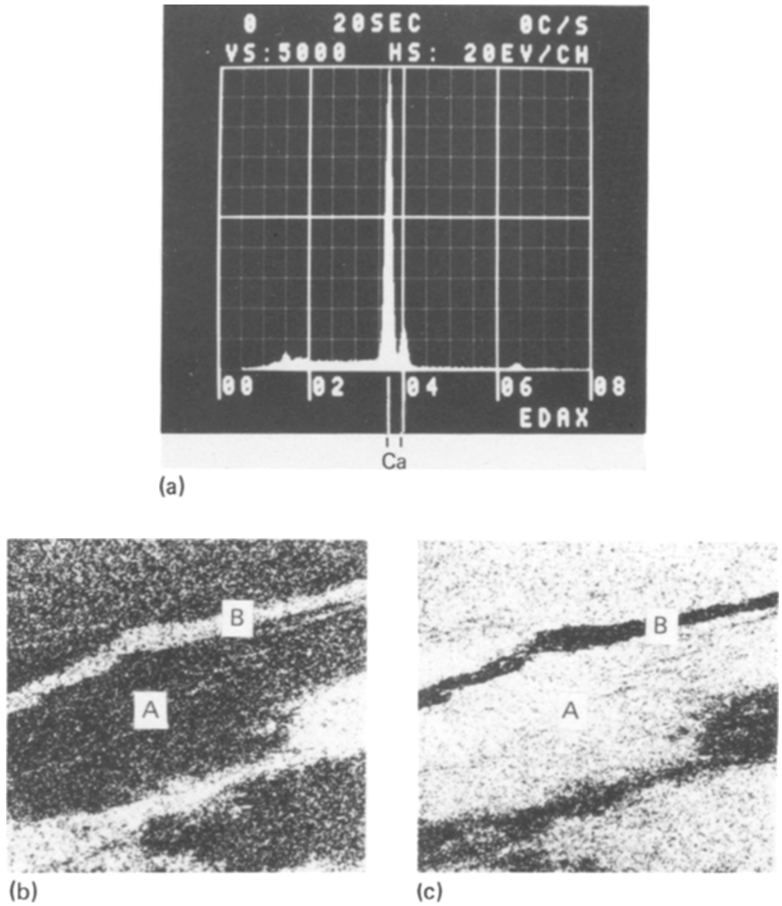

Figure 3 (a) Energy dispersive X-ray analysis of the white vein material in Selva jade. (b) Relative distribution of Ca. (c) Relative distribution of Si. Region A is nephrite and B is a vein.

illustrated in Fig. 1 for the Frazer River jade. The detection of these elements is consistent with the initial identification of the minerals as nephrite. Under similar counting conditions the relative peak heights for Mg, Si and Ca, were the same for each jade. The relative peak heights for Fe were the same in the Russian and Frazer River jades whereas Fe was barely detectable in the Selva jade.

The black inclusions in jade are sometimes called "manganese spots". The X-ray spectra from such inclusions in the Russian and Frazer River jades were essentially the same and that for the Russian material is shown in Fig. 2. The principal elements detected were Cr and Fe, but no Mn. Similar small peaks for Mg, Al and Si were found for inclusions in both jades, with occasional traces of Zn in the black inclusions in the Russian jade. The principal heavy metal in the bright green inclusions in the Russian jade is Cr. Other elements detected were Ca, Si and Mg.

The characteristic spectrum for the white veins in the Selva jade is shown in Fig. 3a. Comparison with Fig. 1 indicates the relative compositional difference between the veins and nephrite, while the relative distribution of Si and Ca in the two regions in Selva jade can be seen in Fig. 3b and c. Although no chemical analysis of the vein material was made, it was thought to be a form of calcium carbonate, since the major element identified from the X-ray spectrum was Ca and also the vein material reacted with dilute hydrochloric acid to produce carbon dioxide.

3.2. Mechanical property measurements

Stable fracture always occurred for all notched and unnotched samples of the three jades tested in four-point bending. The load–time curves recorded on the testing machine had the same general form for all notched samples irrespective of size and material. Fig. 4 shows some examples for notched beams loaded at two rates. Following the initial elastic deflection a small but definite devi-

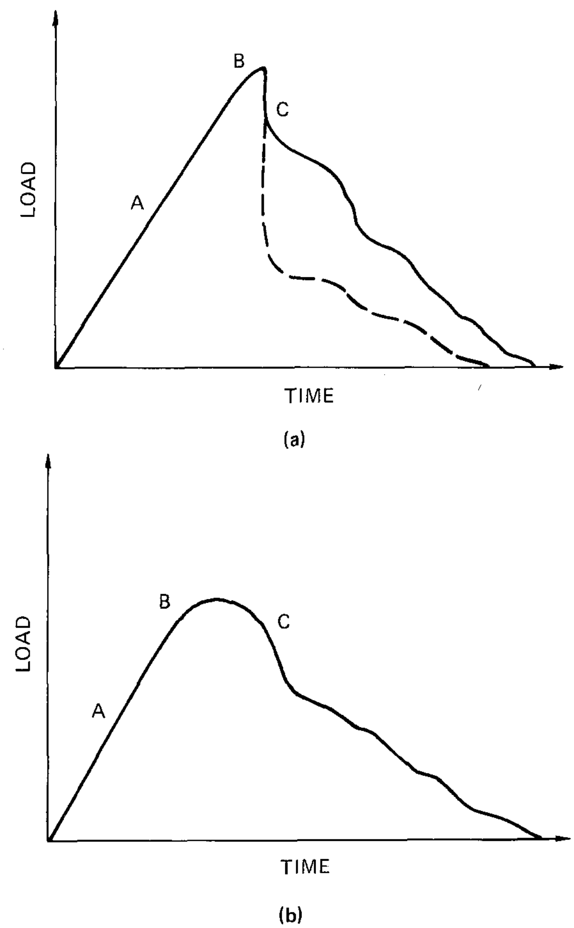

Figure 4 Schematic diagram of load–time curves for jade tested at: (a) 0.05 cm min^{-1}, (b) 0.005 cm min^{-1}. A, elastic region; B, anelastic region; C, region of macroscopic crack propagation. The dashed curve in (a) refers to unnotched specimens.

TABLE I Bend strength of jade

Sample	σMN m^{-2}	Standard deviation	Number of tests
Selva A*	74	10	7
Selva B†	22	6	2
Frazer River	78	23	6
Russian	61	8	7

*Oriented for crack propagation through nephrite islands.
†Oriented for crack propagation through the vein material.

ation from linearity preceeded the fall in load associated with the beginning of macroscropic crack propagation. This anelastic behaviour appears to be similar to that previously reported in tests on rocks [1]. At the higher cross-head speed, 0.05 cm min^{-1}, the initial crack was unstable as indicated by an abrupt change in load as shown in Fig. 4a. With a cross-head rate one decade lower, the initial crack seemed to be stable (Fig. 4b). After this initiation period, the curves for all three jades, at both cross-head speeds, showed a long, continuous tail to zero load as illustrated in Fig. 4. In all cases, cracking was sufficiently stable that a test could be stopped at any time and the sample remained in one piece.

Table I shows values for the strength of jade measured in four-point bending. The strength of Selva jade measured in a direction so that the crack propagated normal to the veins and principally through nephrite islands, is similar to that of the Frazer River material, with the Russian jade being somewhat weaker. Samples of Selva jade cut so that the veins would be in the plane of fracture were very weak and the high incidence of failure of specimens of this orientation during lapping suggested they might contain incipient cracks in these regions.

Values of K_{IC} and G are shown in Table II. Comparing samples of the same size, (6 mm^2 ligament area) the mean value of K_{IC} for the Frazer River jade is considerably higher than that of the other two types. On the other hand, the G values for the Selva and Frazer River jades are very similar but much higher than that of the Russian material. Also included in Table II are results for beams of Selva jade of different dimensions. The larger beams (60 and 200 mm^2) were cut from the same region of the jade block and had a much larger volume fraction of the white veins than in the 6 mm^2 beams. It has been shown previously for rocks [1] that the initial crack length should be relatively long compared with microstructural

TABLE II Fracture toughness and fracture energy of jade

Sample type*	K_{IC} (MN $m^{-3/2}$)	Standard deviation	G(J m^{-2})	Standard deviation	Number of tests	$\frac{\pi^2}{4}\left(\frac{K_{IC}}{\sigma}\right)^2 \times 10^{-3}$ (m)
Selva C	3.6	1.0	562	128	15	5.8
Selva D	2.5	0.6	586	130	21	2.8
Selva E	2.0	0.5	986	161	8	1.8
Frazer River	5.5	1.0	619	127	6	12.3
Russian	2.9	0.4	269	30	12	5.6

*Nominal ligament areas for Selva C, Frazer River and Russian jades were 6 mm^2, with Selva D 60 mm^2 and Selva E 200 mm^2.

features for valid determinations of fracture energy. In the limit, this avoids samples whose ligament size is a single grain, whereby a value of K_{IC} or G which is not representative of the polycrystal can be obtained. In the small Selva beams, the propagating crack rarely interacted with a "grain boundary", i.e. a vein and thus the values are probably representative of the intrinsic ones for nephrite, which depend upon the fine details of the microstructure and which can be compared to the values for the samples from the other two sources. In the larger Selva beams, much of the fracture occurred through vein material and here, the relative sizes of the crack and the two components are very important. In addition, the crack resistance probably depends upon both the macrostructure which determines whether the cracks will run through veins or the islands, and also on the microstructure of the two phases. The fact that different values of G were measured on the 60 and 200 mm^2 beams can be explained on the basis that the macrocrack had probably not developed its full form in the 60 mm^2 beams whereas it is more likely to have done so in the larger beams. On the other hand, the K_{IC} values are not very different and this is because this measurement refers to the initiation of the first unstable crack.

Also reported in Table II are the values for critical flaw sizes calculated from the standard expression derived from the Griffith equation for a penny-shaped crack in an infinite body. The same expression is also used to test the validity of K_{IC} measurements in metallic materials [5]. Since the calculated critical flaw sizes exceed the crack length, a, and the specimen thickness, b, for the 6 mm^2 beams, the fracture toughness values are probably overestimates of K_{IC} and should be considered as K_Q values. However, no standard fracture toughness testing procedures have yet been established for nonmetallic materials, and this is probably the first report of a K_{IC} measurement in a nonmetallic material being questionable on the basis of sample size, while satisfying the microstructural size requirements. For the 60 mm^2 Selva samples, the microstructural criterion might not have been satisfied, as noted above, whereas the critical flaw size is less than a or b. In the case of the 200 mm^2 Selva samples, all requirements are fulfilled to yield a valid K_{IC}.

The initiation of cracks which occur at the maximum load was investigated in large samples (200 mm^2) of Selva jade. In these cases, samples were unloaded immediately following the first reduction in load and the cracked ligament was infiltrated with ink to show up its extent. After some hours, when it was estimated that the ink had fully penetrated and had dried, the samples were tested to failure. In each case the load deflection curves were indistinguidable from those of virgin specimens, i.e. an initial deviation from linearity preceded a well dfined drop in load followed by the normal long tail characteristic of stable fracture. The load at which failure initiated on reloading was closely the same as that at the end of the initial load drop. The extent of the inked region on the fracture surfaces showed that at the first drop in load, approximately half the ligament had fractured. Taking the new crack length as the notch plus the extent of the inked region, the following mean values for four tests were calculated: $K_{IC} = 3.7 \text{ MN m}^{-3/2}$, $G = 1840 \text{ J m}^{-2}$. These values are considerably higher than those measured on uncracked samples (see Table II). Such high values probably arise because of the nature of the crack front. Multiple cracking and crack branching raise the apparent value of K_{IC} and the presence of a large number of cracks, many of which propagate during subsequent fracture leads to the inceased work of fracture.

Attempts to stop tests exactly in the anelastic region, just prior to extensive crack propagation were largely unsuccessful. Although it was supposed that the anelastic behaviour could arise from compliance changes due to the development and opening of microcracks as a precusor to macroscopic crack propagation, no definite evidence for such microcracks could be found. However, during one such test the cross-head was switched off exactly in the anelastic region. The load relaxed slightly then remained essentially constant. On introducing ink into the notch, to detect the crack, a rapid fall in load occurred and the crack proceeded through the complete specimen section, with a corresponding reduction to zero load in about 10 min. The experiment was repeated using distilled water in place of ink and a similar stress corrosion effect was seen. On the other hand, in a further similar test using lapping oil, no load drop or crack propagation occurred, but a subsequent addition of water immediately led to rapid failure.

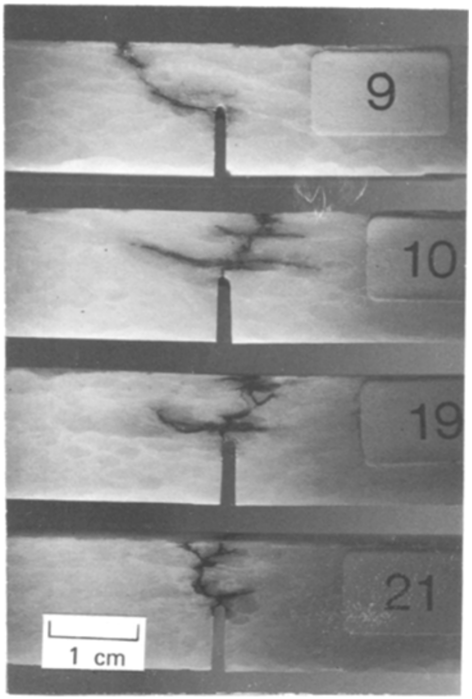

Figure 5 Notched beams of Selva jade (60 mm²) tested to failure. The cracks have been infiltrated with ink to aid identification.

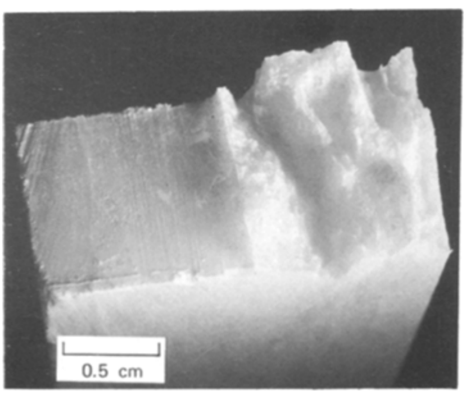

Figure 6 Fracture face in Selva jade.

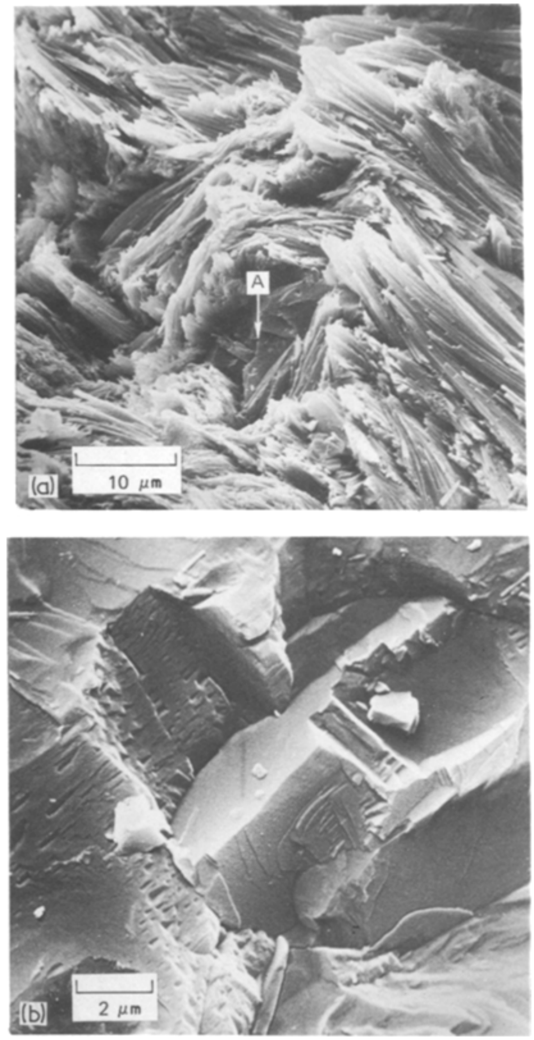

Figure 7 (a) SEM of fractured Selva jade showing nephrite fibres. Note the inclusion of vein material at A. (b) SEM of fracture path through the vein material in Selva jade.

3.3. Fractography

Some examples of fractured samples of Selva jade, infiltrated with ink after testing, are shown in Fig. 5. Crack branching and multiple cracking occurred extensively and some secondary cracks often propagated large distances normal to the crack front. The fracture surfaces were thus highly hackled, as can be seen in Fig. 6, and were always macroscopically much rougher than those of the more homogeneous Russian and Frazer River jades.

In the scanning electron microscope (SEM) two distinct morphologies were seen in the Selva jade as shown in Fig. 7. Energy dispersive X-ray analysis showed that the fibres were nephrite while the plate-like structure corresponded to the vein material. As can be seen from a comparison of Figs. 7, 8 and 9 the nephrite fibres in each jade had a similar appearance. The fibres often occurred as randomly oriented bundles. Both feather-like (Fig. 8) and cylindrical (Fig. 9) bundles were observed and in some regions, the fibres were straight and in others they could be curved and interwoven (Fig. 10). All these possibilities were found in each jade. The fracture surfaces shown in Figs. 7 to 10 show a number of similarities to those of fibre-

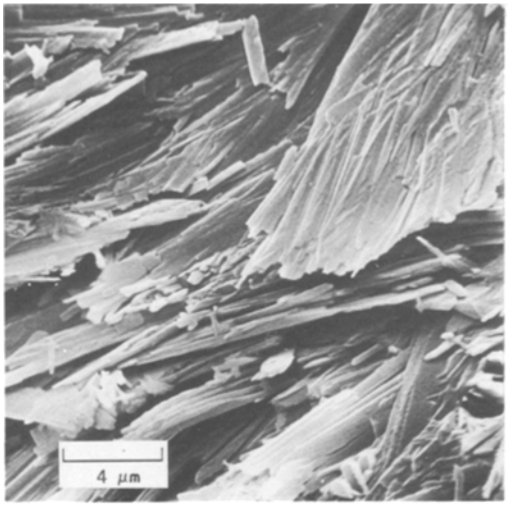

Figure 8 SEM of fractured Frazer River jade showing a region with feather-like fibre bundles.

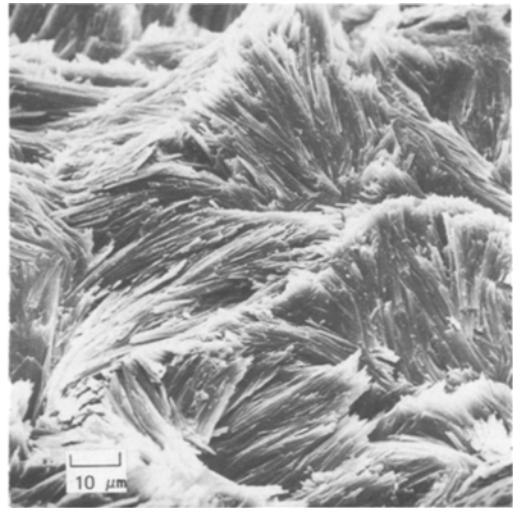

Figure 10 SEM of fractured Russian jade illustrating interwoven nephrite fibres.

Figure 9 SEM showing cylinderical bundles of nephrite fibres, e.g. at A, in Russian jade.

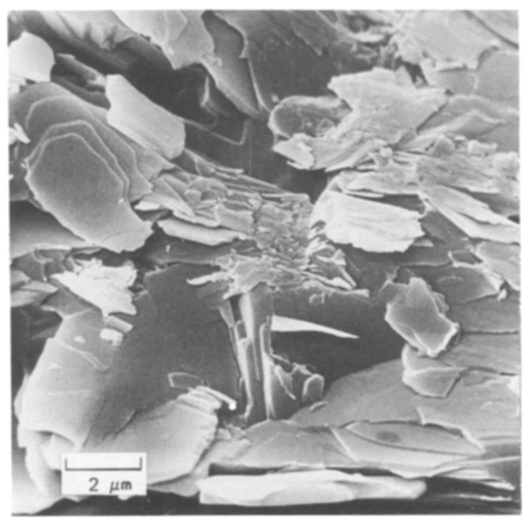

Figure 11 SEM of a fractured black inclusion in Frazer River jade.

reinforced materials, particularly the delamination type failure, i.e. with fracture paths parallel to fibre bundles. This suggests that the strength of bonds between fibres or fibre bundles is less than that of the fibres themselves. In the Selva jade the fracture surfaces of the small notched beams ($6\,mm^2$) and the unnotched beams tested in the high strength orientation were principally fibrous with only a few regions of the plate-like vein material. In contrast, the unnotched beams oriented for crack propagation parallel to the veins showed that the low strength of such beams was associated with easy crack propagation through the vein material. In the Frazer River and Russian jades, the presence of the black inclusions did not have any particular influence on the fracture process. Occasionally, inclusions were seen immediately below a fracture surface suggesting that a path through them was not especially favoured. However, some inclusions were fractured and they had a highly layered appearance as can be seen in Fig. 11.

3.4. General discussion

The fracture resistance of jade is clearly related to the fibrous structure common to the materials from the three sources. The interwoven bundles of fibres are extremely effective in diverting and stopping cracks. Thus, the true surface area created on fracture is very large which explains why G for jade is so high. The differences in K_{IC}, G and σ between the three jades do not appear to be related to the nephrite fibres themselves, since the microstructures are practically indistinguishable from each other, and the fibre dimensions are the same for each jade ($\sim \frac{1}{2}\,\mu$m). Although large flaws of up to 5 mm were detected and avoided in the Russian jade, smaller ones were extremely difficult to observe. Such flaws would be preferential crack paths and a higher density of them in the Russian than in the Frazer River jade would account for the lower mechanical property values found in the former material. The nature of the large flaws was not investigated in detail but in a few samples which were intentionally broken along a predefined natural flaw, the fracture surfaces were indistinguishable from those samples whose failure occurred through unflawed material. The effect of water in promoting subcritical crack growth in jade appears to be similar to that identified as delayed fracture or stress corrosion in glass and various ceramics. Since lapping oil does not cause cracks to grow, it is evident that the ease of cutting or machining thin sections and intricate shapes in jade could be strongly influenced by the coolant used.

Compared to common rocks, such as limestone or granite, jade is an extremely tough material. G values are up to an order of magnitude greater, and although no measurements of K_{IC} for rocks appear to have been made, they might be expected to be very small, in view of the low bend strengths of rocks [1] (0.3 to 8 MN m^{-2}). On the other hand, in comparison with ceramics, like hot-pressed Si_3N_4 or MgO, the bend strengths of jade are relatively low, but again G is 10 to 20 times greater and K_{IC} closely the same. Thus, on the basis of K_{IC} measurements alone, jade is no tougher than most ceramics. However, in jade a fast moving crack which is initially unstable decelerates and becomes stable over a short distance. The present results illustrate clearly the need for specifying the appropriate criterion for fracture. If failure is deemed to have taken place when the first crack is initiated and moves unstably, then K_{IC} appropriately describes this situation. Alternatively, if fracture is taken to mean that the test sample must separate into two pieces, then G, the fracture energy, more accurately describes the process. In ceramics, like silicon nitride, once a crack has been nucleated in a bend specimen, unstable fracture follows by the propagation of this single crack and, in this case the two failure criteria are essentially equivalent.

From the foregoing discusssion, it is apparent that the behaviour of a non-metallic material subjected to a complex loading pattern, such as might arise during machining, cannot be described in terms of a single fracture parameter. Certainly, the reputation for high toughness which jade enjoys from practical experience lies in its fortunate combination of a relatively high K_{IC} coupled with a very high value of G. The advantages of high G, as compared to high K_{IC}, are well known in fibre-reinforced plastics, but no materials have yet been developed with the specific aim of high resistances to crack initiation and to crack propagation. The present investigation of a natural material, jade, indicates that high toughness can be achieved in single phase fibrous non-metallic materials. Although little effort has been made to produce such structures artificially, it may not be too optimistic to hope for medium strength fibrous ceramics with a K_{IC} value around 5 MN m$^{-3/2}$ and G values up to 1000 J m^{-2}.

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