JOURNAL OF MATERIALS SCIENCE 25 (1990) 2170–2174

Strength and toughness of jade and related natural fibrous materials

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Room temperature mechanical properties measured for natural fibrous "jade" materials with random fibre orientations were similar to strengths (e.g. ~100 MPa) and toughnesses, K_{IC}, (e.g. ~3 MPa m$^{1/2}$) in other studies. However, nearly three- and five-fold higher values were found respectively for strength and fracture toughness of "jade" with highly aligned fibres for crack propagation perpendicular to the fibres. Further, the results indicate significantly higher strength and toughness with decreasing fibre diameter and increasing aspect ratio, and an accompanying increase in intrafibre fracture. However, failure was predominantly catastrophic in character for all fibre orientations, indicating some material (i.e. matrix) is necessary for non-catastrophic failure as found in fibre composites.

1. Introduction

Ceramic composites are of increasing interest because of the improved mechanical performance and tailorability of properties achievable in them. Ceramic whisker and especially ceramic fibre composites are of wide interest because of the high toughnesses they can provide, with the non-catastrophic failure obtained in continuous fibre composites being particularly significant. While substantial advances have been made in improving the practicality of processing and in understanding the mechanical behaviour, e.g. toughness, of these materials, substantial further progress is needed. Thus broader understanding of the effects of whisker or fibre volume fractions, lengths and orientation are needed, e.g. whether catastrophic failure is inherent with discontinuous fibre composites as opposed to non-catastrophic failure commonly achieved in continuous fibre composites.

Jade (jadeite and nephrite) and materials of closely related structure and composition such as hornblende are naturally occurring silicates based on aluminium and calcium [1, 2] (Table I). These materials can have varying degrees, sizes and orientations of *in situ* developed fibrous microstructures. Further, both anisotropy and substantial levels of fracture toughness (3 to 8 MPa m$^{1/2}$) are believed to be due to the fibrous nature of the microstructure of these materials [3, 4]. Table I summarizes these earlier mechanical studies. The *in situ* formation of these fibrous microstructures, the essentially 100% resultant fibre structure and resultant mechanical behaviour make these useful materials of study.

This paper reports results of a study of the mechanical properties of these materials that complements and extends earlier studies of similar materials [3, 4].

2. Experimental procedures

Three different samples of commercial "jades" were selected (Table II), some after sample examination of their microstructures, from a local lapidary store. Two of these are designated by their claimed origins, Siberia and Guatemala. The third is designated LB hornblende based on its commercial origin since its geographic origin is unknown. X-ray diffraction analysis revealed that one of these materials (Guatemalan) was jadeite and two were hornblende as shown in Table II. Microstructural examination showed one of the materials (LB) to have a high degree of preferred orientation of the fibres as well as very fine fibres, while the other two appeared to have overall a random orientation of fibres. There were also a range of fibre characters, e.g. the Guatemalan jadeite had large (e.g. ~20 μm diameter), blocky (e.g. aspect ratio ~2) grains, the Siberian hornblende had finer (e.g. ~5 μm diameter) grains with higher aspect ratios and the LB hornblende had still finer (~1 μm diameter) grain with much higher aspect ratios. The irregular pieces were first cut into blocks in which sound wave velocities were measured by the pulse-echo method [5]. Young's modules and Poisson's ratio were then calculated from these velocities using density data obtained by Archimedes' method. In order to evaluate possible or expected affects of fibre orientation on toughness and strength, specimens were diamond machined from the three samples in three orthogonal directions, Fig. 1, (i.e. so in the case of LB hornblende material the fracture surfaces would be parallel or perpendicular to the highly oriented fibres).

Double cantilever beam (DCB) toughness specimens machined from these blocks were ~12 mm × 40 mm × 3 mm with a centre groove of roughly half

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TABLE I Properties of jade and related materials

Material	Jadeite	Nephrite	Hornblende
Crystal Structure	Monoclinic (2/*m*)	Monoclinic (2/*m*)	Monoclinic (2/*m*)
Formula	$NaAl(SiO_3)_2$	$Ca_2(Mg, Fe)_{5(OH)(Si_4O_{11})_2}$	$Ca_2(Mg, Fe)_5(OH)_2(Al. Si)_8O_{22}$
Density[a] ($g\,cm^{-3}$)	3.3–3.5	2.9–3.3	2.9–3.3
Cleavage plane[a]	(1 1 0)	(1 1 0)	(1 1 0)
Hardness[a] (Moh)	6.5–7	5–6	5–6
Young's modulus (MPa)	132[b] 205[c]	222[b]	115–155[c]
Flexural strength (MPa)	105[b]	217[b] 21–77[d]	
Fracture toughness ($MPa\,m^{1/2}$)	7[b] 6.4[c]	7–8[b] 2–6[d]	3.4[c]
Fracture energy ($J\,m^{-2}$)	120[b] 94[c]	200–1000[b] 500–2000[d]	49[c]

[a][1, 2]
[b][3]
[c]This study (without "L" arms)
[d][4]

of the thickness of the specimen (~ 1.5 mm) to guide the crack propagation. In the case of the LB hornblende, which had a definite fibre orientation as noted above, the blocks and resultant specimens were oriented relative to the fibres as shown in Fig. 1.

The applied Moment Double Cantilever Beam (AMDCB) technique [6] was utilized for measurement of the critical stress intensity factors, K_{IC}, of these materials. Centre grooved specimens were pre-cracked and pivot arms epoxy bonded for loading in a mechanical test machine*. Due to high toughness and/or textured nature of some of the specimens, epoxied pivot arms suffered debonding from the specimen in some cases while other specimen fractured sideways by delaminating along the fibres. To overcome these problems, L-shaped specimen holding arms [7] were used in conjunction with the AMDCB measurements. These L-shaped arms are designed both to withstand higher applied load (more than 200 kg in some cases) and to reinforce the specimen arms. Some AMDCB specimens were examined by microradiographic techniques described elsewhere [8] as well as in transmitted light optical microscope to reveal actual crack characters. Resultant fractured halves of these DCB specimens were retouched to finish the rough fracture edges and used as flexural strength specimens.

Three-point flexural tests were used to measure the strengths using a span of 12 mm and a crosshead speed of $1\,mm\,min^{-1}$. All mechanical tests were carried out at ~ 22° C and nominally 50% relative humidity. Scanning electron micrographs (SEM) were obtained for the fractured surface to analyse the microstructure of these materials.

3. Results

Initial fracture toughness results (without the use of "L" arms), Table I, are consistent with other available studies, as is the range of Young's moduli found. However, as noted earlier, the number of successful K_{IC} tests without the "L" arms was very limited (typically 1 to 3 values per material) and orientation effects were not considered in these initial studies. Results of more detailed studies including orientation effects using the "L" arms are shown in Table II. The fracture toughness of Guatemalan jadeite and Siberian hornblende are similar in both absolute values and in not showing much orientation dependence. On the other hand, much higher toughness and a pronounced orientation dependence was observed for the fine fibre, highly oriented hornblende of unknown geographic origin, i.e. the LB hornblende, with the highest toughness being observed for crack propagation perpendicular to the fibre direction. It should be noted that some variations of toughness was measured for

TABLE II Mechanical properties of jade and related materials

Material (origin)	Density ρ ($g\,cm^{-3}$)	Elastic modulus (GPa)	Poisson's ratio	Specimen type*	Toughness K_{Ic}( )† ($MPa\,m^{1/2}$)	Flexural strength (MPa)†
				A	3.1 ± 0.9(5)	
Jadeite (Gautemala)	3.3	205	0.256	B	2.6 ± 1.3(6)	107 ± 9(6)
				C	3.6 ± 1.4(5)	
				A	8.2 ± 1.5(3)	288 ± 32(8) (A, B)
Hornblende (LB, unknown)	3.4	151	0.197	B	7.2 ± 1.1(4)	
				C	16.2 ± 5.3(4)	40 ± 19(8)
				A	3.9 ± 1.2(7)	
Hornblende (Siberian)	3.0	117	0.175	B	3.7 ± 0.2(4)	93 ± 13(8)
				C	3.5 ± 1.5(4)	

*See Fig. 1.
†Number of specimens tested.

*Instron Universal Testing Instrument, Model 1122, Instron Corporation, Canton, Massachusetts, USA.

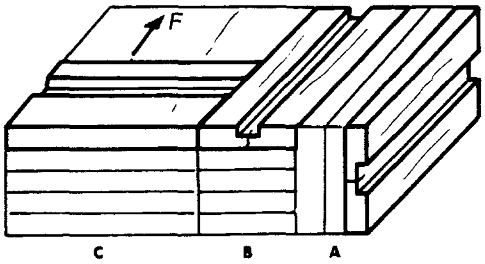

Figure 1 Schematic diagram of how specimens for both toughness and strength were machined from a block (of LB hornblende having highly aligned fibres) such that the cracks propagated either perpendicular or parallel to the fibre orientation (arrow *F*).

each materials depending on the location from which the specimens were machined. Fig. 2 shows a DCB specimen examined by microradiography while under load, revealing multiple and tortuous cracks. Fig. 3 shows a transmitted light optical micrograph of a "jade" sample with the fibres oriented approximately perpendicular to the crack propagation direction, again illustrating the complexity of crack propagation.

Flexural strengths were again similar for Guatemalan jadeite and Siberian hornblende and showed no orientation dependence. However, much higher flexural strengths were measured for the fine fibre oriented LB hornblende for A and B specimens, i.e. for fracture propagating perpendicular to the fibre direction and substantially lower for C specimens, i.e. for crack propagation parallel with the fibre direction. The failure of the flexure specimens was generally catastrophic (Fig. 4). Thus all high strength specimens showed essentially an instantaneous drop of over 80% of the load upon failure; only the relatively weak LB hornblende C specimens (i.e. for the tensile direction perpendicular to the fibre direction) showed retention of a significant percentage of their maximum load carrying capacity after failure. Specimens typically broke into two pieces or were usually readily separated into two pieces by hand after testing. However, resultant fractures were quite irregular, commonly involving a significant amount of approximately laminar failure mode, mainly for the strong-tough orientation of the LB hornblende (e.g. Fig. 5).

Figs 6 to 11 are SEM micrographs of the fracture surfaces showing the fracture and the microstructure of the materials studied. Fig. 6, for the Guatemalan

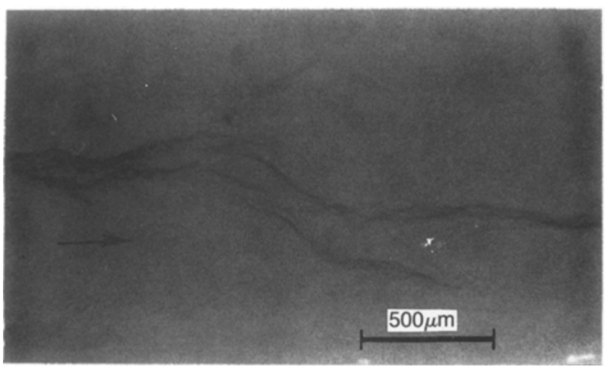

Figure 2 X-ray microradiography of crack propagation in Guatemalan jadeite while under AMDCB test showing multiple and tortuous cracks.

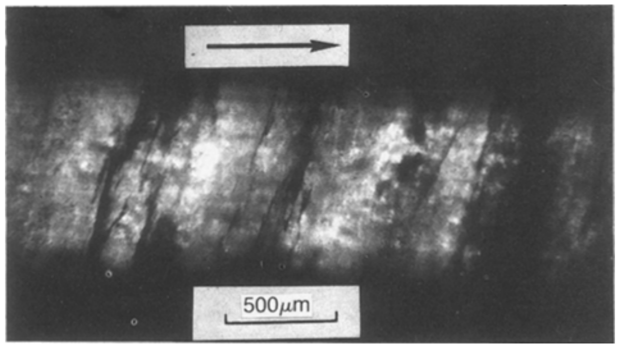

Figure 3 Transmission light optical micrograph of LB hornblende specimen showing aligned fibres and associated cracking perpendicular to the intended crack propagation direction (arrow).

jadeite, besides showing the generally random orientation of the generally short, coarse fibres, shows essentially completely transgranular fracture. Higher magnifications of many grains shows intersecting cleavage planes (Fig. 7) while variable and difficult to discern, fibre diameters were estimated at $\sim 20 \mu m$ and aspect ratio of ~ 2.

Fracture surfaces of the Siberian hornblende, also revealed a generally random fibre orientation, Fig. 8. Higher magnifications showed occasional small regions of aligned fibres, Fig. 9, again showing predominantly transgranular fracture. However, the fibres were generally thinner (e.g. $\sim 5 \mu m$ diameter) than those for the Guatemalan jadeite and exhibited a somewhat flaky fracture.

Fracture surface of the LB hornblende besides illustrating the much greater fibre alignment relative to the above two materials, showed a much more pronounced fibrous nature (i.e. high aspect ratio of the order of 100), and much finer fibre diameters (e.g. $\sim 1 \mu m$). Fracture of the LB hornblende involved substantial delamination along the fibre length, Fig. 10.

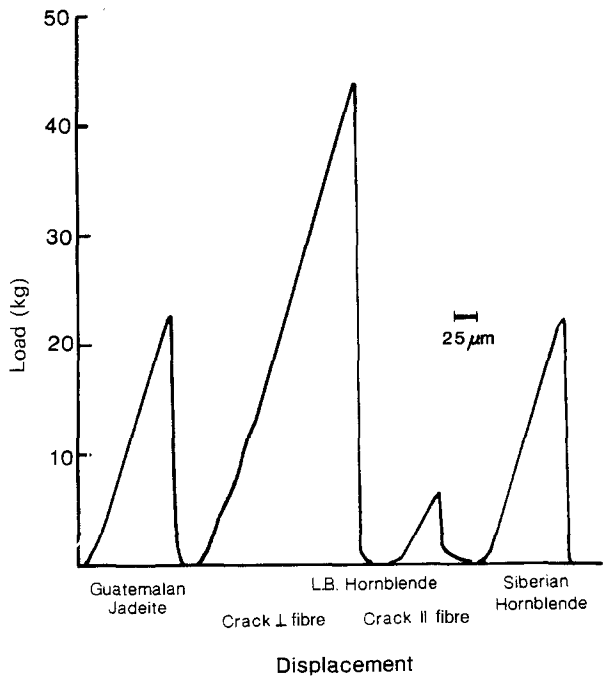

Figure 4 Load–displacement curves showing the failure of specimen in the flexure strength test were, in general, all catastrophic.

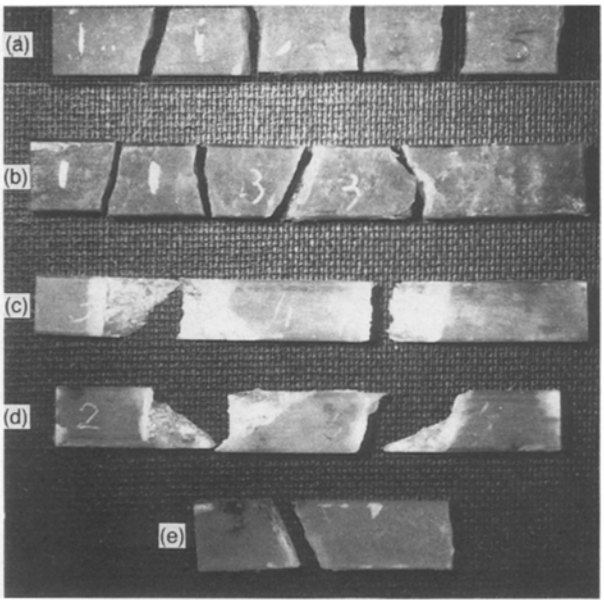

Figure 5 Fracture of flexural test specimens showing the irregular, partially laminar failure found, especially for LB hornblende specimens. (a) Guatemalan jadeite, (b) Siberian hornblende, (c) LB hornblende A, (d) LB hornblende B, (e) LB hornblende C.

4. Discussions

Overall the present results for the Guatemalan and Siberian jades are similar to those of Bradt, Newman and Biggers [3], and of Rowcliffe and Fruhaff [4]. In particular, Young's modulus and strength are similar despite difference in compositions, Tables I and II. Variations in mechanical properties are clearly due to sample variations, e.g. as directly observed in this study. However, this study shows substantial dependence of these properties on fibre orientation and diameter which are likely to be major factors beyond compositional (e.g. purity) factors. The effect of fibre orientation and associated anisotropy in turn introduces variations since these materials do not fully satisfy the fracture mechanics assumptions of most of the K_{IC} tests. Thus, two materials of this study had at least some local macro-anisotropy (e.g. Figs 6 and 8). K_{IC} results are also reflected in the tortuous nature of the crack observed in varying degrees in all three materials studied. Examination of the more detailed results of this study reveal that microstructure is a major factor in their mechanical properties.

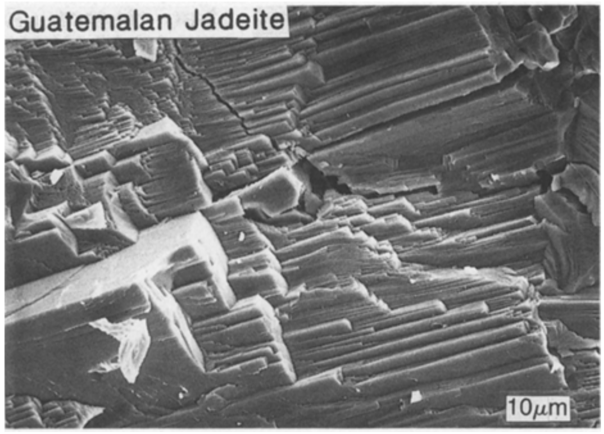

Figure 7 Cleavage (i.e. transgranular) fracture of Guatemalan jadeite.

Consider first the effect of microstructural orientation shown in this study. The Siberian hornblende and Guatemalan jadeite, where overall the fibres are generally randomly oriented have relatively isotropic flexural strengths and toughnesses (Table II), as observed in other studies [3, 4]. On the other hand, the fine fibre, highly oriented LB hornblende shows distinctly anisotropic flexural strength and fracture toughness. Crack propagation, parallel to the generally aligned fibres, results in significantly lower toughness and strength than for crack propagation normal to the fibre orientation, i.e. similar to unidirectional fibre composites.

The results also show the highest strength and toughness is associated with the finest fibre (grain) diameter and greatest fibre length (i.e. highest aspect ratio). Thus the average stength and fracture toughness of the fine fibre, highly oriented LB hornblende are each about twice those of the other two "jades". More detailed comparison of the data suggests this fibre (grain) size (i.e. diameter–length, aspect ratio) trend may be a basic one, at least for fracture toughness and energy. Thus, the Siberian hornblende clearly has intermediate K_{IC} values and intermediate fibre (grain) diameter and length. Further, recognizing the lower Young's modules of the Siberian hornblende, it would have about twice the fracture energy of the Guatemalan jadeite, reinforcing the indicated effect of (fibre) grain diameter.

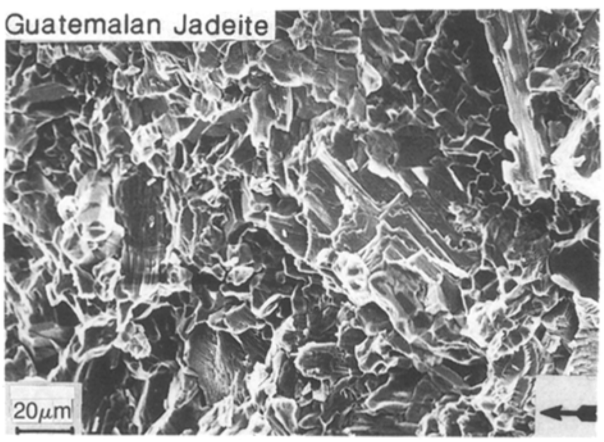

Figure 6 Fracture surface of a Guatemalan jadeite showing predominantly transgranular fracture mode, and general microstructure.

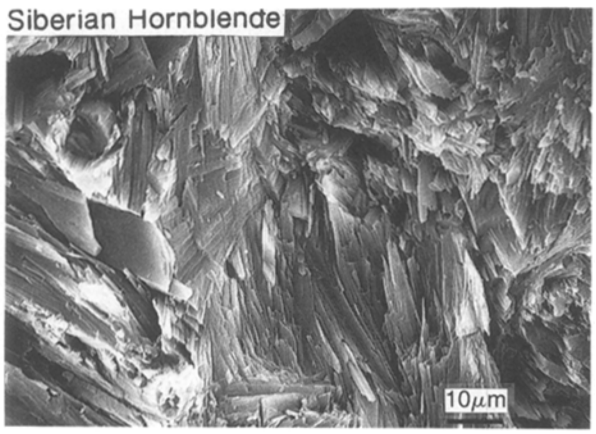

Figure 8 Overall fibre character and random fibre orientation observed in Siberian hornblende.

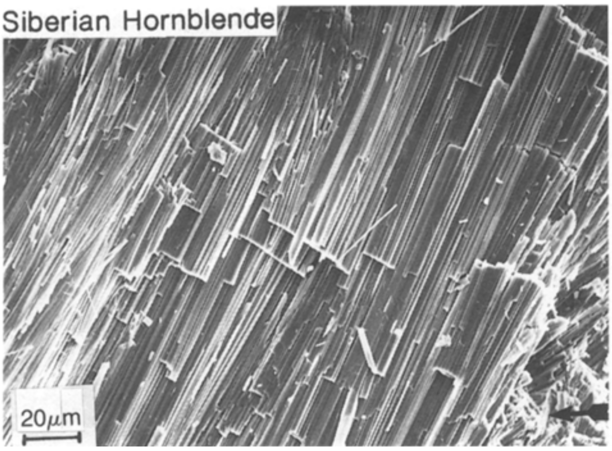

Figure 9 Regions of aligned fibres in Siberian hornblende. Also note the predominant transgranular fracture.

Figure 10 Fracture surface of LB hornblende showing much more pronounced fibrous nature and high degree alignment of the fibres.

The high toughness found for these materials generally correlated with their strengths (e.g. compare results for the LB hornblende specimens of different orientations, Table II). However, high toughness did not correlate with non-catastrophic failure, in fact the opposite; i.e. the toughest had the most catastrophic failure and the weakest ones the least catastrophic failure (i.e. a greater retention of load carrying capability past the maximum load sustained) Fig. 4. This correlation of the degree of catastrophic failure with strength is not uncommon, i.e. specimens of low strength often exhibit incomplete failure simply because they lack enough store mechanical energy for complete failure. However, the essentially catastrophic failure of the high strength material with such high toughness (and related fibrous structure) is somewhat surprising. This may indicate that the high toughnesses obtained from macrocracks are not applicable on the microscale of fracture, e.g. as suggested for other systems [9]. However, this high toughness–catastrophic failure dichotomy appears to imply that some level of matrix is needed between fibres in a fibrous body (i.e. composite) in order to have non-catastrophic failure.

Thus, while the high toughness of these materials is clearly related to their fibrous character, i.e. fibre diameter and aspect ratio, as discussed earlier, basic questions remain. Clearly, composition and related fibre crystal structure are basic issues. Both the toughness and related complex crack character clearly are very similar to fibre composites where limited bonding between fibre and matrix are central to good toughness [10]. However, the absence of extensive non-catastrophic failure in these jade-type material which are with essentially all fibres and very little, or no, matrix is a fundamental difference from such fibre composites, thus raising issues of the microstructural aspects of their toughness, strength and failure. Thus basic questions are: (1) to what extent and how is the toughness due to the quite anisotropic crystal structures (e.g. monoclinic) and the morphotropic character of the fibrous grains e.g. the role of difference in properties (e.g. Young's modulus and thermal expansion) as well as intrinsic bonding between grains determine toughness, and (2) whether the affects of crack–microstructure scales on toughness, e.g. the use of large cracks that can multiply branch in toughness tests, is applicable to smaller cracks that control strength? Understanding of these affects could be of major benefit to further development of fibre composites. It could also be of major value in developing other ceramics since there should be numerous structural analogues of jade type materials with more refractory capabilities and hence of wider practical use than jade materials.

5. Summary and conclusions

This study further demonstrates that naturally fibrous "jade" materials can have high toughness, that this toughness is significantly affected by fibre character and is associated with complex, tortuous, and multiple cracks. Fracture toughness and especially fracture energies increase with decreasing fibre (grain) diameter and increasing length (i.e. aspect ratio). However, while strength generally correlated with toughness, non-catastrophic failure clearly did not. Despite the high toughness and very fibrous microstructure, catastrophic failure generally occurred. Determining the source of this toughness and its relation to failure characteristics can be of significant value in developing ceramic fibre (and whisker) composites and in developing expected more refractory analogues of jades.

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Received 3 January
and accepted 23 August 1989