Origin and development of flexibility in asbestiform fibres

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Abstract

Quantitative measurement of relative flexibility have been made on amphibole fibres based on calculation of bending strain of maximally bent fibre segments. Enhanced flexibility was found to be an inverse function of fibre diameter. Asbestos is therefore a morphological entity. These conclusions are supported by consistent observations of fibre diameter/flexibility relationships in amphiboles, serpentine, and rutile. Flexibility may arise from growth of thin crystals (primary flexibility), or from crystal diameter reduction (secondary flexibility). Mechanisms for secondary flexibility in amphiboles include splitting and solution along cleavage planes. Gross morphological characteristics are a poor indicator of microscopic habit, since coarsely fibrous minerals such as picrolite antigorite may not be fibrous after particle width reduction, and secondary asbestiform fibres are often derived from minerals with a non-fibrous morphology on gross examination.

KEYWORDS: asbestos, flexibility, fibre, tremolite, actinolite, rutile, asbestiform, carcinogenesis.

Introduction

IT has been suggested that flexible mineral fibres possess some unique chemical structure by virtue of which flexibility arises, and that this unique structural chemistry is associated with carcinogenesis. However, careful study has failed to show any consistent differences in chemical composition or structure between the asbestos varieties and their non-asbestiform analogues (Zoltai, 1979, 1981; National Research Council, 1984), and work in experimental carcinogenesis has supported the hypothesis that mineral fibre carcinogenicity is related to dimensional characteristics of fibres (Stanton *et al.*, 1977; Brown *et al.*, 1979; Cook *et al.*, 1982). These findings suggest that flexibility and carcinogenesis are not caused by some special geochemical attribute of asbestos fibres, and that both are related to dimensional characteristics.

Recently it has been proposed that flexibility results from the properties of a surface layer which possesses greater strength than inner structures (Zoltai, 1979, 1981; National Research Council, 1984). The existence of such a surface layer has been demonstrated in a wide variety of solids, including the asbestos minerals (Zoltai, 1979, 1981; Griffith, 1921). In this study, a threshold maximum width for the occurrence of flexibility is described, below which flexibility increases sharply. This provides evidence for the surface-layer model of asbestos flexibility, since specific surface area (surface area/volume) increases with decreasing width.

The relationship between fibre width and flexibility proposed here is borne out by observations we have made of asbestiform mineral occurrences at numereous locations in the northern New Jersey area (Germine and Puffer, 1981; Germine, 1985), including serpentines, amphiboles, fibrous clays, zeolites, and rutile. Our observations indicate that flexibility arises not only in thin fibres bounded by crystal growth surfaces but also in fibres with borders produced by cleavage and solutional processes. In this paper, we will describe some representative samples illustrating these concepts.

Methods and procedures

The analytical standards and methods used for mineral identification have been previously described (Germine and Puffer, 1981; Germine, 1985). The tremolite standard is from South Korea (Rohl *et al.*, 1976). Scanning electron microscope energy-dispersive X-ray spectroscopy (SEM-EDXS) was used to obtain chemical analyses of amphibole samples, and identification was made on the basis of these data using the standard nomenclature (Leake, 1978) together with X-ray

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diffraction and optical data. Electron diffraction under transmission electron microscopy (TEM) was used for further amphibole characterization. Serpentine minerals were identified using a combination of criteria (Mumpton, 1974), including optical, X-ray diffraction, and TEM characterization. The characteristic morphology of chrysotile under TEM was used for identification in mixed serpentine samples (Puffer *et al.*, 1987).

Determination of flexibility

Flexibility in minerals is defined as the ability of a particle to bend without breaking and without resuming its original shape (A.G.I. Glossary of Geology, 1977). Elasticity refers to the ability of a mineral fibre to resume its original shape after bending. In actual usage flexibility is reserved for substances which undergo marked bending under short-term and mild to moderate stress. The stress that is exerted in bending a substance is, by definition, proportional to bending strain, which, in fibres, is directly proportional to a/r, where a is the fibre diameter and r is the radius of a circular fibre segment, or torus segment, which forms around the point of greatest bending stress (Hearle *et al.*, 1969).

Relative flexibility was determined by measuring circular fibre segments under PLM. Use of PLM permitted direct observation of the bending properties of fibres. Individual fibres were bent using a probe while immersed in oil on a glass slide, and allowed to recoil slowly to a point at which a circular segment of minimum radius was maintained without external pressure. The fibres were then photographed, and measurements of fibres were made from photographs using a measuring magnifier at a total magnification of approximately 1000×. Measurements were calculated against a micrometer slide, and the radius of the torus was calculated using a computer program that provides the radius given the length of sides of the inscribed triangle. The index of strain, a/r, is, under the experimental conditions described, proportional to relative flexibility.

Results and conclusions

To facilitate description and discussion of findings, results will be presented by examination of flexibility in single crystals, flexibility in fibre aggregates, and flexibility secondary to alteration. As will be demonstrated, flexibility in aggregates involves both the characteristics of individual fibres and cohesion between fibres. Such fibre aggregates are formed by binding together of individual thin crystals, by separation of thicker crystals into thinner fibrous units along cleavage surfaces, or, most commonly in the amphiboles, by a combination of these processes. Based on the relationship to crystallization history, we will refer to flexible fibres consisting of single crystals as possessing 'primary flexibility', and to flexible fibres split from larger crystals as possessing 'secondary flexibility'.

Flexibility in single crystals. Asbestiform minerals most commonly occur in fibre aggregates. Because it is difficult to isolate individual fibres, such aggregates cannot be readily used to determine flexibility in single crystalline units. Therefore, samples containing individual fibres, each representing a separate crystal, were utilized to describe this relationship.

Actinolitic amphibole from the Palisades Sill, Fort Lee, New Jersey (Germine and Puffer, 1981) consists of web-like masses of individual crystals. The composition of this amphibole, as determined by energy-dispersive X-ray analysis (Table 1) is approximately intermediate between that of actinolite and actinolitic hornblende. The sample analysed contains fibres ranging in width from less than a micron width to 7 μm or more. Fibres thinner than 3 μm show marked bending which is not observed in thicker fibres (Fig. 1).

Table 1. SEM-EDXS analyses of selected samples*

	1	2	3	4
SiO_2	51.0	48.8	58.5	49.1
Al_2O_3	-	3.7	-	-
FeO	36.4	23.8	1.3	10.0
MgO	7.5	7.6	18.4	14.9
MnO	1.9	-	-	-
Cr_2O_3	-	-	-	3.8
CaO	-	13.2	18.6	17.2
Na_2O	-	-	-	-
K_2O	-	1.6	-	1.8

1. IARC grunerite asbestos.
2. Actinolitic amphibole, Fort Lee, New Jersey.
   Ions per molecule base on 23(O):
   Si 7.18, Al 0.65, Fe 3.20, Mg 1.66, Ca 2.07, K 0.15
3. Tremolite, Forks, Pennsylvania.
4. Actinolite, Franklin, New Jersey.

*Corrections for atomic number, atomic weight, and fluorescence effects were done using the ASAP program. Analyses have been normalized to an anhydrous total of 96.8%.

The results of analysis of relative flexibility in the Fort Lee actinolitic amphibole are shown in

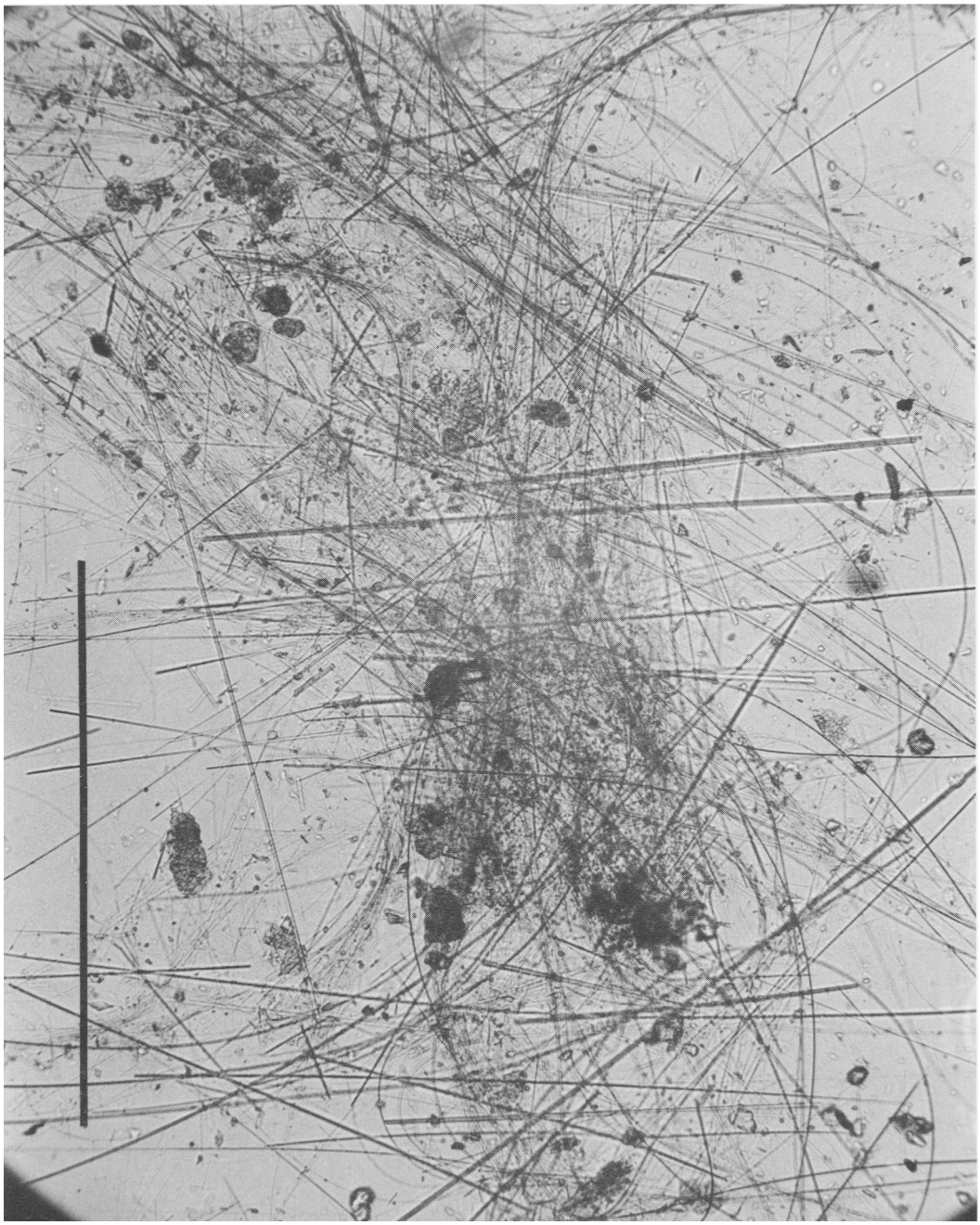

FIG. 1. Actinolitic amphibole from Fort Lee, New Jersey. Note semicircular fibre profiles, which provide an index of strain in bent fibres. Bar = 0.5 mm.

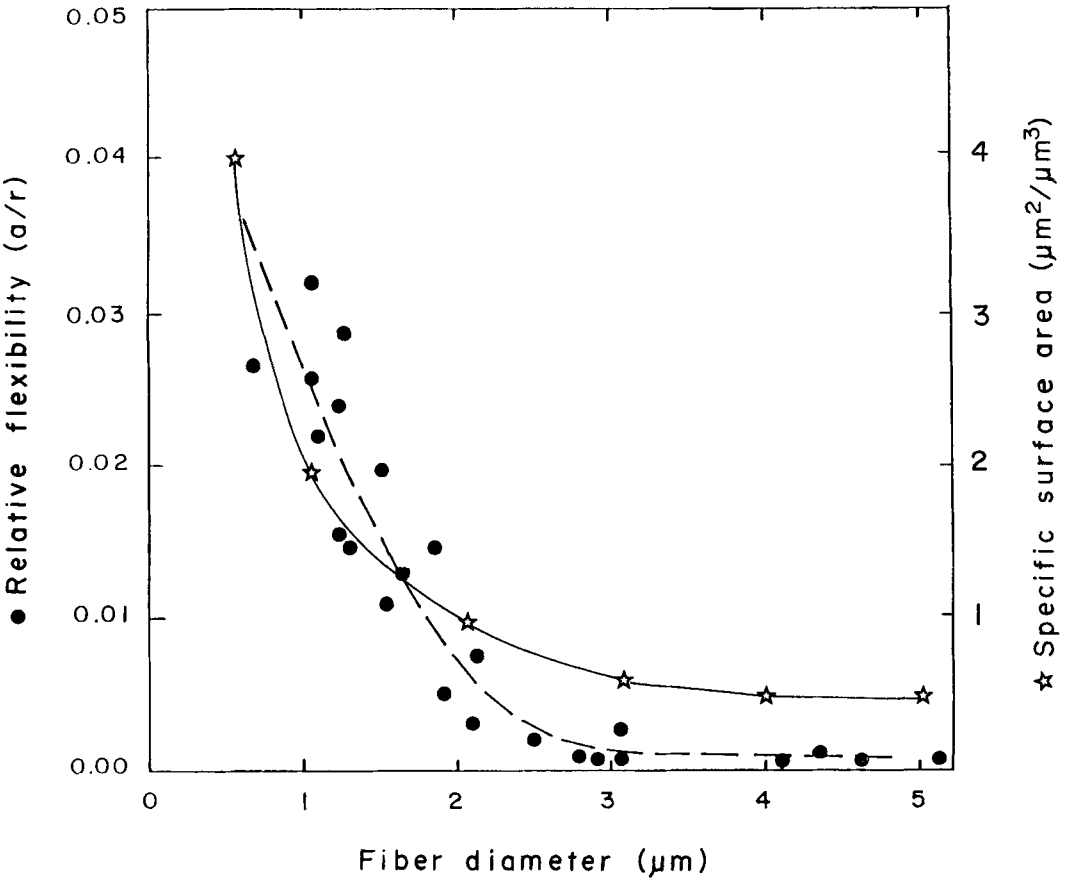

FIG. 2. Graph showing relationship between relative flexibility and fibre diameter in actinolitic amphibole fibres from Fort Lee, New Jersey. Also shown is relationship between specific surface area and fibre diameter.

Fig. 2. Also shown is the approximate specific surface area of fibres, assuming cylindrical profiles. The thicker fibres exhibit a consistently low relative flexibility. Markedly enhanced flexibility begins to appear at about 3 μm, consistent with qualitative observations. There is a several-fold increase in relative flexibility below this 'threshold' diameter.

Flexibility in single crystals is a direct reflection of tensile or bending strength (Zoltai, 1981). Both chrysotile and amphibole have been shown to increase sharply in strength in fibres with diameter less than 3 to 4 μm, with strength increasing several times between diameters of 4.0 and 0.2 μm (Zoltai, 1981). This increase in strength is consistent with the thresholds for enhanced flexibility at widths of 3 to 4 μm reported here for actinolitic amphibole from Fort Lee.

Fig. 2 shows the nearly parallel relationship between specific surface area and relative flexibility. Specific surface area is equivalent to the reciprocal radius of the fibre, which has been previously related to strength in whiskers and synthetic amphibole fibres (Zoltai, 1981). Our data clearly extend this observation to natural asbestos fibres, underscoring the relationship between flexibility and strength, and providing strong evidence for the surface layer model of flexibility (Zoltai, 1981). These results show that not only must the surface layer have high tensile strength, but must be able to accommodate strain energy so that fibres remain bent and do not recoil elastically. The inflection in the flexibility diameter curve at about 3 μm does not appear to result strictly from increased strength, but rather from a transition from elastic to flexible behaviour,

since what is actually being measured is the radius of curvature after recoil.

The relationship between flexibility and fibre width is not restricted to fibres formed by crystal boundaries, but extends to fibres produced by splitting of larger fibres along cleavages. Upon grinding, Fort Lee actinolitic amphibole fibres tend to break into thinner fibres along cleavages, and TEM examination of the crushed sample shows that these fibres exhibit marked bending, and, in some instances, frayed or splayed ends. Measurements of the radius of curvature of such fibres using TEM indicate that the minimum radius has a relationship to fibre diameter that approximately follows the curve shown in Fig. 2 in the submicron range. The Fort Lee amphibole as well as other fine 'byssolite' type fibrous amphiboles tended to show very prominent bend contours which migrated up and down along fibre length in complex patterns under electron beam exposure (Germine and Puffer, 1981, Fig. 4*b*). This feature was also observed, although less marked, in asbestiform rutile. Some byssolite samples, upon size reduction by grinding or in weathering profiles, produced a preponderance of irregular, non-fibrous or low aspect-ratio fibrous fragments. TEM observations suggest that density of diffraction contrasts is related to production of fibre on grinding of such byssolite-type fibres, and such contrasts were particularly prominent in ground fibres exhibiting splayed ends.

The development of primary flexibility is not restricted to the 'asbestos minerals', i.e. chrysotile and the amphiboles. Rutile (TiO_2), which is chemically and crystallographically dissimilar to these minerals, occurs in leathery masses of flexible fibres at Franklin, New Jersey (Germine, 1985; Puffer *et al.*, 1987). Flexible fibrous masses are composed of crystals which typically range from 0.07 to 0.10 μm. Twinned fibres are not infrequent. The material possesses a pronounced fibril-bundle structure, in spite of the fact that the crystals are free-growing, and therefore have no distinct preferred orientation. Upon grinding, fibrils tend to break across fibre length.

Flexibility in fibre aggregates. A common characteristic of asbestiform mineral fibres is their occurrence in bundles. Such bundles possess aggregate flexibility that is not unlike flexibility in rope. In ropes, flexibility is determined by the ease with which frictional forces are overcome between adjoining fibres, thereby allowing fibres to slip relative to one another during bending operations (Himmelfarb, 1957). Slippage between mineral fibres allows each fibre to behave as an independent unit. Since strain at a given radius of bending is proportional to the width of the fibre (Hearle *et al.*, 1969), strain on bending in fibre aggregates through a given radius will decrease with fibre separation, leading to enhanced aggregate flexibility. Therefore, the flexibility in bundles or aggregates of fibres arises in two ways: (*a*) from the flexibility of individual fibres, which depends on their diameter; and (*b*) from the degree of aggregation which depends on the weakness of bonding between adjacent fibres.

In asbestos minerals, such as chrysotile and crocidolite, which typically occur in parallel aggregates of extremely fine crystals, fibril separation can often be accomplish by mechanical means, as has been employed in the asbestos industry. Tremolite, actinolite, and anthophyllite, however, more commonly occur as coarse fibrous crystals which are comparatively brittle even when separated. Varieties of these minerals that are analogous to crocidolite are comparatively rare. For example, among the numerous occurrences of asbestiform tremolite that we described in the northern New Jersey area (Germine and Puffer, 1981), there was only one rather limited occurrence at Sparta, N. J., where ultrafine crystals were characteristic. This tremolite showed diffuse, parallel extinction due to random orientation of sub-microscopic tremolite crystals. In all other samples (more than 100 from several locations) extinction was characteristically inclined, and individual crystalline units were well into the range of observation by light microscopy. Further reduction of fibre width occurred along cleavage or parting, and was often observed to involve weathering. In some samples fibres which have been separated by weathering assume a more-or-less random orientation, leading to diffuse low-angle or parallel extinction, and greatly increased flexibility. In these cases we say that flexibility, which is only poorly developed in the relatively coarse crystalline units, is secondarily enhanced. Again, the reciprocal increase in flexibility with decreasing fibril diameter is clearly in evidence and is expressed by differential movement of fibres in aggregates.

Tightly bound mineral fibre aggregates exhibit little flexibility. When such fibres are separated by mechanical means or by chemical alteration processes, flexibility is markedly enhanced. This phenomenon explains previous reports of increased flexibility in amphibole fibre aggregates after weathering (De Toit, 1945; Ladoo and Meyers, 1951; Rice, 1957; and Germine, 1986). Weathering enhancement of flexibility in amphibole is well demonstrated in samples of asbestiform tremolite from Forks, PA (Table 1). Fresh material is coarse, splintery, and relatively brittle. With weathering, and as the material moves up

the soil profile, the fibre becomes silky and highly flexible, eventually degrading to a mixture of montmorillonitic clay and very fine asbestos fibre.

Individual tubular fibrils of chrysotile were always observed to be less than 1 μm in diameter, and as such, exhibit primary flexibility, although aggregate flexibility may be absent in tightly bound, interlocking fibrils (Germine and Puffer, 1981). In one fibrous variety of serpentine, picrolite antigorite, flexibility is generally absent. Fresh samples of fibrous antigorite from Staten island, New York, are splintery and relatively brittle. Individual fibrils generally range in width from 7 to 24 μm, and when ground to submicron size, the material is predominantly non-fibrous under the TEM. Upon weathering and as the material moves up the soil profile, fibrils and fibril aggregates separate, and the material takes on marked aggregate elasticity. Aggregates can be bent and twisted through more than 360°, but recoil and resume their original shape when pressure is released. The elastic properties of individual fibrils are expressed through slippage between fibrils. However, since individual fibrils are above the threshold width of enhanced flexibility, aggregate flexibility is absent. When the fibrils are mechanically disaggregated, a small proportion of fibrils are found with widths less than 4 μm, and these fibrils exhibit flexibility as expressed in bending.

Flexibility secondary to alteration of non-fibrous minerals. Development of flexibility by fibre-width reduction was noted in samples of amphibole from the Franklin marble in Franklin and Sparta, New Jersey, where prismatic crystals of tremolite, actinolite, and edenite are hydrothermally altered in zones to a mixture of fibrous amphibole, and variable amounts of talc (Germine, 1986). Edenite is a member of the hornblende group (Deer *et al.*, 1963, p. 209), and as such is not among the regulated asbestos minerals. Chromian actinolite (Table 1) was also noted in a secondary asbestiform variety at Franklin.

In all cases, the amphiboles retained the external prismatic or acicular form of the amphibole prior to alteration. Thus, the outward form is actually a kind of pseudomorphism, as becomes particularly apparent as the process of fibre genesis proceeds. This process has also been noted in ferroactinolite from Minnesota by T. Zoltai (pers. comm., 1985), who describes the ferroactinolite used by Cook *et al.* (1982) in an experimental carcinogenesis assay as being of this type.

Alteration occurs on the periphery and along cleavage traces of amphibole crystals. Individual tightly bound polyfibrillar aggregates begin to demonstrate flexibility between widths of 3 and 4 μm. Upon weathering, this amphibole, which is otherwise relatively brittle and tightly bound, develops into highly flexible fibre. Fibres first separate into coarse units, and then progressively into finer fibrils, with progressive development of flexibility and fibril-bundle structure (Fig. 3). Often lenticular or cavernous etch pits develop as part of this weathering process (Fig. 3), as previously noted in pyribole weathering (Berner *et al.*, 1980). Individual crystals sometimes show areas that display marked longitudinal etching, and produce flexible fibres apparently bordered by solution surfaces, as well as areas which produce more irregular fragments. The same process was also observed in alterations of clinopyroxene to amphibole (Germine, 1981).

TEM, SEM, and thin-section observations indicate that alteration results in a very marked cleavage, usually on (110). TEM orientations indicate that (100) surfaces are involved as well, as previously noted in calcic amphibole cleavage (Dorling and Zussman, 1987). In the samples of tremolite and anthophyllite that show the most marked secondary flexibility, cleavage or parting tends to occur principally on (100). Electron diffraction observations indicate that this involves either multiple twinning on (100) or intergrowth with talc. The (100) cleavage appears to be a talc-like cleavage that is most marked in talc-amphibole intergrowths of the type described by Veblen *et al.* (1977), although it also appears in the absence of talc, and, significantly, becomes more marked in sub-micron particles. This talc-like cleavage is particularly marked in slip fibre, and appears to be the principal surface for translation between fibres.

TEM observations of ground tremolite samples show that the weathering process is accompanied by accentuation, and perhaps production *de novo*, of (010) diffraction contrasts. Weathering origin of chain-width disorder, which produces such contrasts, has been observed by Eggleton and Boland (1982). These well developed diffraction contrasts are associated with development of very fine fibrils down to a width of less than 500 μm which are highly flexible and occur in bundles much like chrysotile. Orientation within aggregates becomes increasingly random, but aggregates otherwise retain the electron diffraction characteristics of the original tremolite.

Discussion

Measurements of the relative flexibility of asbestiform mineral fibres have been made, and it has been found that flexibility increases as the diameter falls, with an inflection at about 3 μm. This variation is parallel with the variation in ten-

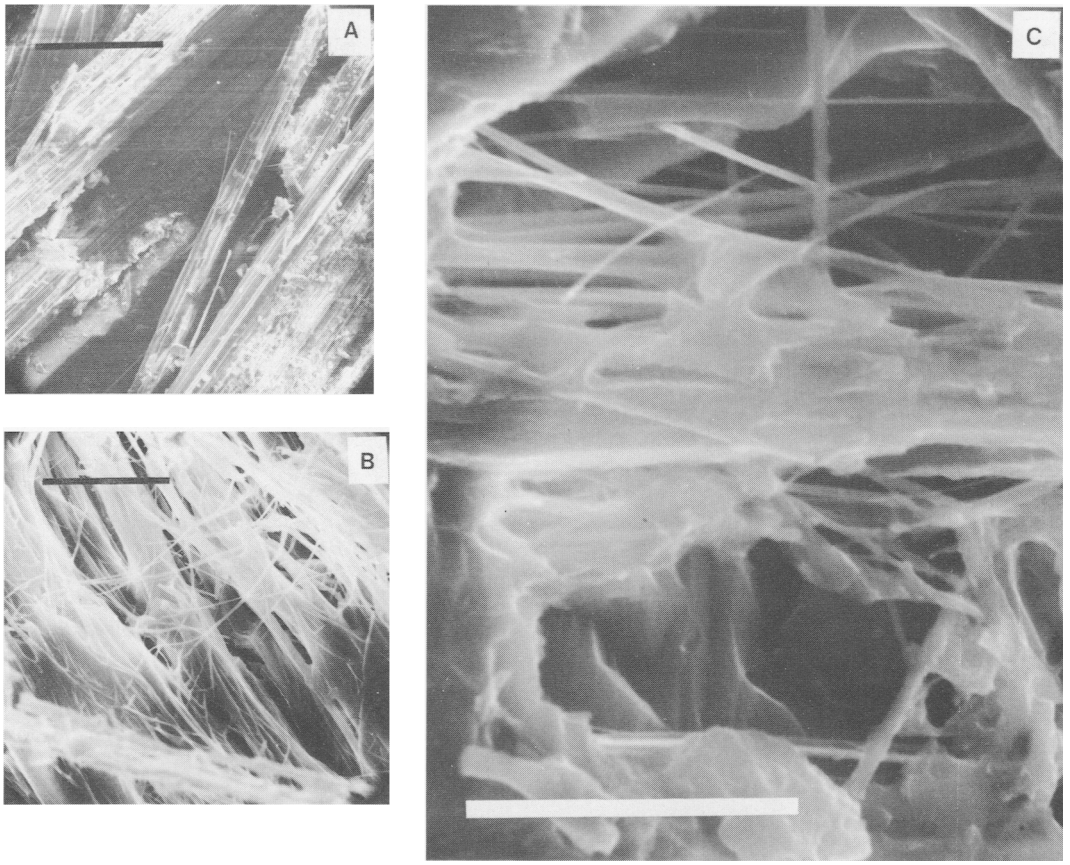

FIG. 3. SEM photographs of grossly prismatic tremolite showing development of secondary asbestiform habit microscopically. A: Specimen taken from a euhedral tremolite crystal measuring approximately 3 cm × 0.5 cm, showing transition from strongly-preferred cleavage to fibrillar structure. Note bending of loose fibre. Bar equals 100 μm B: Highly developed secondary asbestiform texture with fibre separation associated with weathering. Note solutional features, loss of parallel fibre orientation, and production of thin, flexible fibres. Bar equals 10 μm. C: Interior of large solutional etch pit showing intergrowth of fibrous and non-fibrous amphibole and talc. Bar equals 10 μm.

sile strength with diameter. The inflection appears to mark a transition from elastic to flexible behaviour. In addition to the quantitative measurements of flexibility, we have described observations on a wide range of asbestiform fibres with surfaces produced by a variety of processes, and all seem to conform to a qualitatively similar flexibility–diameter relationship.

Flexibility has not been previously well documented in the hornblende-group minerals (Campbell *et al.*, 1977; Zussman, 1979). It has been proposed by Zussman (1979) that substitution of Si by Al in the hornblende group brings about a preference for growth of prism faces, thereby preventing formation of highly elongated crystals. The pressure and temperature conditions under which aluminum substitution occurs are also likely determinants of crystal habit. It is therefore highly significant that the material identified here as transitional between flexible and non-flexible fibres is also transitional between actinolite and actinolitic hornblende. It is also significant that secondary flexibility is documented here in a hornblende group mineral. This shows that it is morphology, rather than composition, which prevents members of the hornblende-group from exhibiting primary flexibility.

The same hydrothermal alteration that leads to enhanced splitting along cleavage planes also enhances or potentiates the process that can lead

to isolation of thin, markedly flexible fibres by weathering processes. It is well known that hydrothermal solutions gain access to mineral substrates along cleavage planes, and, as shown here, these cleavage planes become a nidus for alteration. Therefore, splitting along cleavage planes in secondary asbestiform amphibole is not the same as cleavage in primary amphibole. It follows an internal fibrillar structure which has been produced by alteration. This is seen not only in secondary asbestiform amphibole but also in primary asbestiform amphibole with secondary enhancement of fibrillar structure and therefore flexibility. The relative permeability of such primary fibrous amphiboles, their structural occurrence in veins which are conduits for hydrothermal solutions, and the probability of retrograde alteration in the wake of hydrothermal solutions involved in primary crystalization, all favour this type of secondary enhancement, making it, in our observations, a very common phenomenon.

The observed relationship between fibril width and flexibility provides evidence for the view that flexibility is dependent on structural properties near the surface of mineral fibres (Zoltai, 1981). The enhanced strength of the surface layer has been well-documented and measured in all the minerals discussed here. The results of our study provide evidence that this enhanced strength is the major determinant in mineral fibre flexibility. The increased specific surface area in thin fibres would then account for their flexibility. Such an explanation would also be consistent with the observation of flexibility in platy minerals such as chlorite, talc, and graphite. The occurrence of flexibility in these minerals and a non-silicate structure such as rutile, and the consistent observation of markedly enhanced flexibility in fibres less than 3 to 4 μm in diameter in a wide range of samples, suggest very strongly that surface area is the major controlling variable in relative flexibility within mineral groups. Specific internal structural variations, such as chain width disorder (Veblen and Buseck, 1980; Veblen *et al.*, 1977), appear to be linked to fibre splitting, but may not, in themselves, be directly related to flexibility in mineral fibres.

Our observations indicate that external or gross characteristics are a poor indicator of microscopic habit. Such characteristics have been frequently used in mineralogical characterization of asbestiform and non-asbestiform minerals (Zussman, 1987) and studies involving characterization of habit for environmental health interpretation (Campbell *et al.*, 1977) In the latter application, particularly, it is important that microscopic habit be carefully described, since health effects are related to microscopic particle characteristics and not to the external form of the particle source. In this regard, use of the term asbestiform for fibres of high aspect ratio, in the sense that the term had been traditionally used by mineralogists (Zussman, 1977), is more appropriate than use of terminology that stresses gross or bulk properties of commercial asbestos. There do not appear to be clear-cut analytical criteria for identifying asbestos as defined commercially, and our observations extend those of Dorling and Zussman (1987) in this area. Secondary asbestiform amphiboles may not exhibit characteristics of primary asbestiform amphiboles such as twinning and fibre growth boundaries, while primary asbestiform amphiboles need not possess chain-width disorder characteristics that are present in some secondary asbestiform amphiboles. Production of thin, highly elongated particles on comminution appears to be the only essential characteristics of flexible mineral fibres, and may involve a variety of mechanisms.

The fact that secondary asbestiform amphibole has been shown to be more highly carcinogenic than primary asbestiform amphibole suggests that *in vivo* fibre splitting is an important mechanism in carcinogenesis, since it increases the tissue burden of long, thin fibres (Cook *et al.*, 1982). This process appears to be analogous to the fibre splitting we have observed in a weathering environment. Workers in the environmental health field should be alerted to the existence and potential hazards of such secondary asbestiform amphibole, rather than being given the mistaken impression that it is benign because of its gross prismatic or acicular form. Such amphiboles require careful study, including microscopic assessment of particle size and aspect ratio characteristics and alteration characteristics, before assessment of potential mineral fibre hazard can be properly performed.

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