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Deformation-induced volume damage in particulate-filled epoxy resins

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In this investigation the microstructural deformation and damage mechanisms in an epoxy resin filled with silica flour are investigated. The unfilled resin exhibits bulk shear yielding in both compression and tension which is localized into fine bands by the addition of the particulate filler. In addition, the filled material stress-whitens in compression and at lower rates and higher temperatures in tension. The shear yielding is recoverable upon heating for a time above the glass transition temperature; however, the stress whitening is irreversible. The addition of filler particles introduces several damage mechanisms including particle–matrix debonding, particle fracture and matrix microcracking. In the following report, these damage mechanisms are characterized microstructurally in terms of the rate, temperature and type of loading and their relative contributions to stress whitening.

1. Introduction

Rigid particulate fillers are added to epoxy resins to reduce costs and improve mechanical properties. For example, the addition of rigid particles increases Young's modulus, the yield strength and the toughness of epoxies. The majority of investigations of these composites have been mechanical in nature [1–9]. There have been no thorough investigations of the microstructural behaviour of these materials in response to deformation. In spite of this, a great deal of information about the microstructural deformation mechanisms has been inferred from these studies and, on occasions, some supporting microstructural evidence has been presented.

It is now generally accepted in the literature that shear yielding is the primary deformation mechanism in epoxy resins [1, 3]. In the past, there has been some disagreement as to the occurrence of crazing in highly crosslinked polymers; however, recent work by Glad [10] has shown conclusively that crazing is not a microdeformation mechanism in highly crosslinked epoxies. Thus crazing has not been addressed in the current investigation.

In a purely mechanical analysis, Young *et al.* [3] found that the inclusion of particles (rigid or soft) in an epoxy resin alters the yield strength and the yield strain. The fundamental mechanism of deformation, which is shear yielding in the matrix, was unchanged by the presence of particles. They did not present any microstructural evidence of shear yielding in this work. In terms of physical characterization of shear bands, there is no work in the literature concerning epoxy resins. This can partly be understood in consideration of the general intractability of these materials. In thermoplastic materials, however, shear bands are well characterized [11–14].

A number of authors have reported the occurrence of particle–matrix interfacial adhesion failure (i.e. debonding) in filled epoxy composites. Smith *et al.* [15] measured the volume expansion behaviour of glass bead-filled epoxy as well as the stress–strain behaviour. They proposed that debonding was a gradual process, beginning at low strains and continuing past the peak strain into the region of strain-softening. They supported this mechanical analysis by showing micrographs of debonded particles on the fracture surfaces. Evans *et al.* [16] attributed the non-linearity in the deformation behaviour of a glass bead-filled epoxy to debonding and matrix microcracking between particles. They presented microscopic evidence from Owen [17] showing debonding in the plastic zone at the tip of a slow moving crack.

The only reported microstructural evidence of deformation-induced volume damage in a silica particulate-filled epoxy resin is that by Young and Beaumont [18]. They performed compression tests and were able to show that particles in the deformation zone were fractured in a plane parallel to the compression direction. Furthermore, they mentioned that debonding could be generated on a fracture surface by a very slow-moving crack.

Kinloch *et al.* [19] presented microscopic evidence of the plastic zone at the crack tip of a rubber–glass-bead–epoxy hybrid system. In addition to cavitation of the rubber phase, they reported debonding of glass beads and shear bands in the matrix. Shear yielding was inferred from dark lines or furrows running between particles ahead of the crack tip. They

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suggested, like others [2, 20], that the glass beads promote shear yielding in a manner similar (but less efficient) to that of rubber particles; however, the presence of the rubber particles in the hybrid makes it difficult to prove.

Stress whitening is a discoloration of the material in response to deformation. It is due to the scattering of light from free surfaces such as crazes in thermoplastics or cavitation in rubber-modified epoxies. Stress whitening has been observed in silica-filled epoxies after yield in compression experiments [18], but no explanation for its occurrence has been given.

As a result of the above investigations, a number of deformation and damage mechanisms have been identified in rigid particulate-filled polymers. These include matrix shear yielding, particle–matrix debonding, particle fracture and microcracking in the matrix. Each mechanism has been proposed separately by various authors and analysis has mainly relied upon mechanical testing. Although there is some microstructural evidence for these mechanisms, they have not been treated together and characterized in the context of deformation-induced volume damage.

In this investigation the microstructural behaviour of an unfilled and a silica-filled epoxy resin in response to deformation is examined. The purpose of the work is to determine the cause of stress whitening and to identify and characterize microscopically the types of damage mechanism occurring in these materials.

2. Experimental procedure

The epoxy used in this investigation was composed of a hydantoin-based resin and an acid anhydride hardener with a glass transition temperature of approximately 125° C. The filler was silica flour with a mean particle size of 50 μm. For most investigations the filler volume fraction was 42%; however, a 16% mixture was used for more detailed studies of shear bands. Specimens were cast in a vacuum and cured for 4 h at 80° C and then 16 h at 130° C.

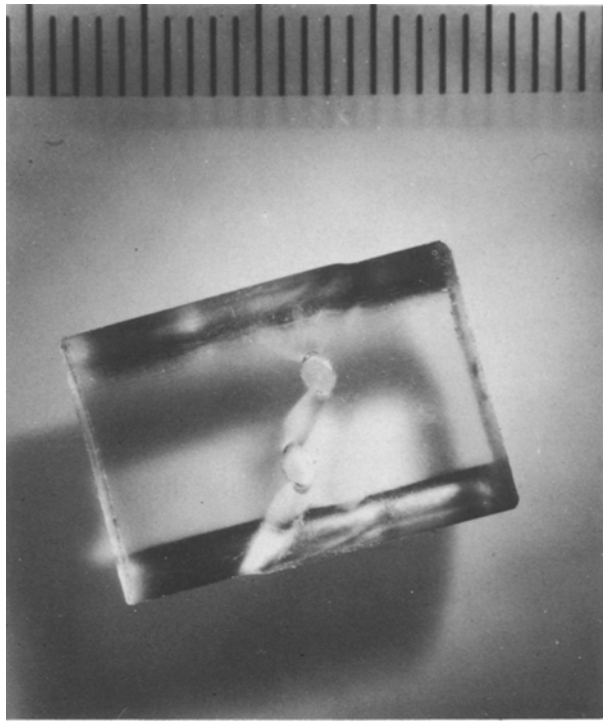

Figure 2 Shear bands in a compression specimen of unfilled resin tested at 23° C. Note the classic shear offset at the edges. The shear yielding was localized by holes drilled in the specimen prior to deformation. Specimen diameter is 11 mm.

Cylindrical tensile specimens with a gauge length of 80 mm and a diameter of 11.2 mm were cast in steel moulds. Cylindrical compression specimens with a 1.5 aspect ratio were cut from the gauge sections. A lathe was used in order to ensure a perpendicular cut. Specimens were lubricated with silicone disulphide grease and compressed in an Instron testing machine equipped with oven and temperature control.

Tensile strength tests were performed at rates of 0.05 to 50 mm min^{-1} at temperatures of 23, 50, 85 and 105° C. Compression tests were done at the same temperatures but at rates of 0.5 and 5.0 mm min^{-1} only. A limited number of creep rupture experiments were

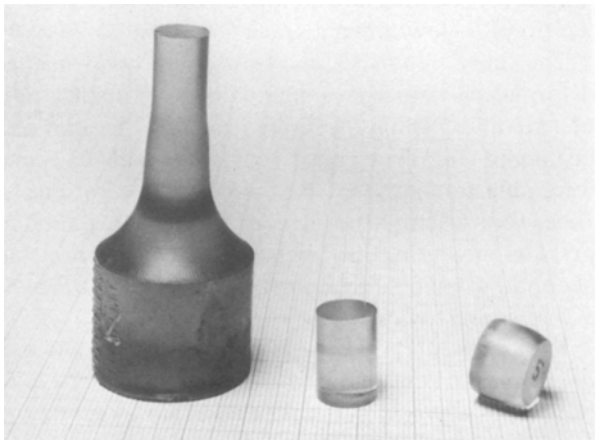

Figure 1 Examples of bulk shear yielding in neat resin. The left specimen is a uniaxial tensile specimen tested at 105° C, in the centre is an undeformed compression specimen and at the right a uniaxial compression specimen compressed at room temperature. The original diameter for these specimens is 11 mm.

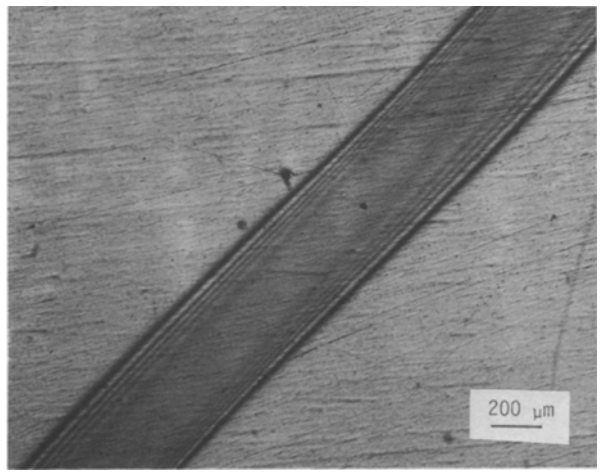

Figure 3 Birefringent shear band in the neat resin tested in compression at room temperature and viewed in transmitted polarized light.

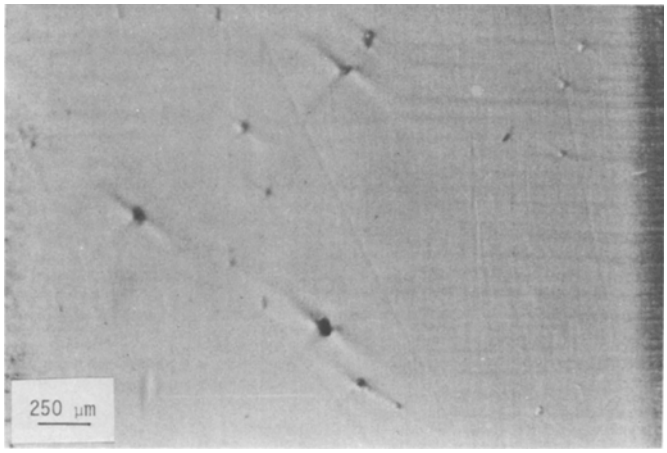

Figure 4 Shear bands in the neat epoxy resin initiated by inhomogeneities on the surface of a creep specimen at 85° C.

performed at 85 and 105° C on shoulder-bar specimens with a square cross-section.

The primary aim of this investigation was to observe volume damage in these materials and thus it was necessary to section specimens for observation. Thin sections were prepared following the methods of Holik *et al.* [21]. Essentially, the specimen to be examined was cast into a block of epoxy and cured at room temperature. Once cured, the block was cut and polished parallel to the surface to be observed. This was then glued to a non-birefringent Plexiglass slide with an optically transparent adhesive. Another cut was then made parallel to the slide surface, leaving a thin slice of material adhering to the slide. This section, now supported by the slide, could be ground and then polished. The resulting thin section could be observed in transmitted and reflected light.

Scanning electron micrographs were taken using a Cambridge Instruments S100 electron microscope. Fracture surfaces of specimens were coated with gold and observed at 15 kV. Transmission optical microscopy was performed on a Lietz optical microscope equipped with polarizers.

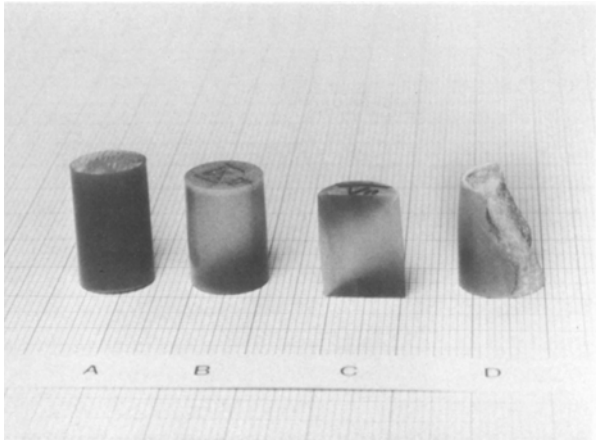

Figure 5 Examples of compression damage in 42% material: (A) undeformed specimen, (B) deformed specimen showing stress whitening, (C) same as in (B) sectioned longitudinally to show deformation zone (white area) and the cone-shaped dead zones, (D) specimen deformed to failure showing fracture at the dead zone–deformation zone interface.

Polishing such two-phase materials with a 30-fold difference in moduli between the phases could give rise to artefacts. It was thus necessary to verify that both the debonding of particles and the particle fracture observed on polished surfaces were not artefacts of polishing or other preparation techniques. Concurrent work on fracture in these materials has shown [22] that at a given temperature there is a maximum speed at which a moving crack produces a debonded surface. At higher speeds the crack propagates in the matrix, unless there is previous damage to the material. In this investigation specimens were compressed and then fractured transversely in impact. These surfaces were compared with undeformed impact fracture specimens and also those prepared by polishing, in order to evaluate any damage created by the polishing method.

3. Results

3.1. Shear yielding in the unfilled resin

In spite of the predominantly brittle behaviour of epoxy resins, considerable ductility may be obtained when brittle failure is suppressed. In compression, bulk shear yielding of pure resin was obtained at all temperatures investigated (23 to 105° C). An example of such yielding at 85° C is shown in Fig. 1, where an undeformed specimen is compared with a specimen deformed in uniaxial compression. These specimens exhibit barrelling as well as some buckling at higher strains. At room temperature compressive strains of over 15% are attainable. Ductility is, of course, extended by increased temperature and lower rates of loading.

In tension, limited ductility may be attained at the higher temperatures and under creep loading. A typical example is shown in Fig. 1 where the specimen has necked prior to brittle failure (105° C). Strains obtained in tension are considerably lower than those in compression and the material is highly sensitive to even the slightest flaws.

Localized yielding, specifically shear banding, occurs whenever, and wherever, stress inhomogeneities are present. Fig. 2 shows shear bands in a compression specimen induced by holes drilled in the specimen prior to deformation. These bulk shear bands produce the classic shear offset at the edge of the specimen. Such a shear band, viewed in transmitted polarized

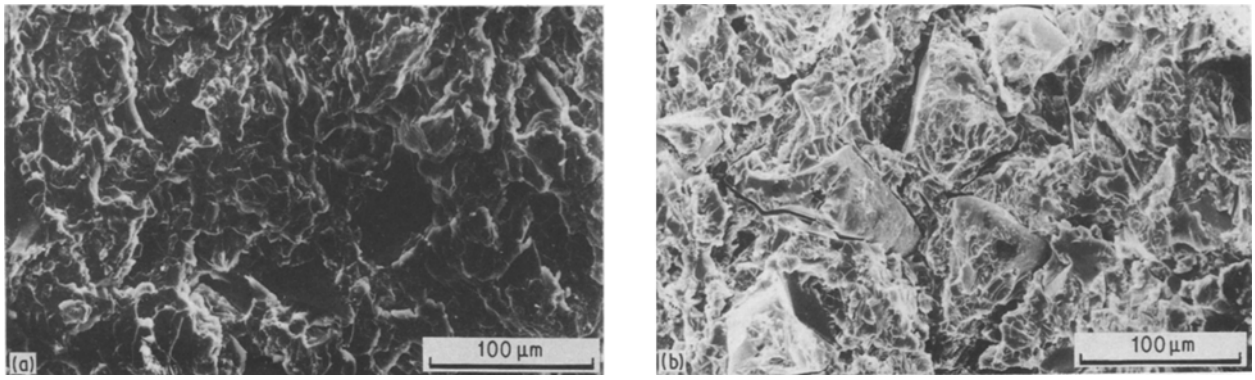

Figure 6 SEM micrographs of fracture surfaces in the 42% material created by impact fracture of damaged and undamaged material. (a) Undeformed specimen, with particles undamaged and well bonded to the matrix. (b) Specimen compressed uniaxially until significantly whitened and then impact-fractured perpendicular to the principal stress. Many fractured and debonded particles are visible.

light, is shown in Fig. 3. The coarse band is approximately 45° to the principal stress. The size of the band seems to depend on the size of the heterogeneity at which it originated and the strain. In tension, shear bands often initiate at inhomogeneities; see for example Fig. 4 where shear bands initiated at foreign inclusions can be seen on the surface of a tensile specimen.

Shear yielding in the unfilled epoxy, whether in bulk or localized form, was found to be recoverable upon heating above the glass transition temperature of a few hours.

3.2. Compressive damage in filled resins

Filling the epoxy resin with mineral particles such as silica flour enhances the stiffness of the composites and changes other properties [2]. The ductile flow exhibited in compression of above 15% strain (despite a fracture strain under 2% in tension) cannot occur without the accumulation of damage.

The first obvious signs of damage occur at the yield point where the stress–strain curve changes slope. At this point the material begins to stress-whiten, indicating the initiation of damage. Whitening occurs in a diffuse band that runs diagonally across the specimen.

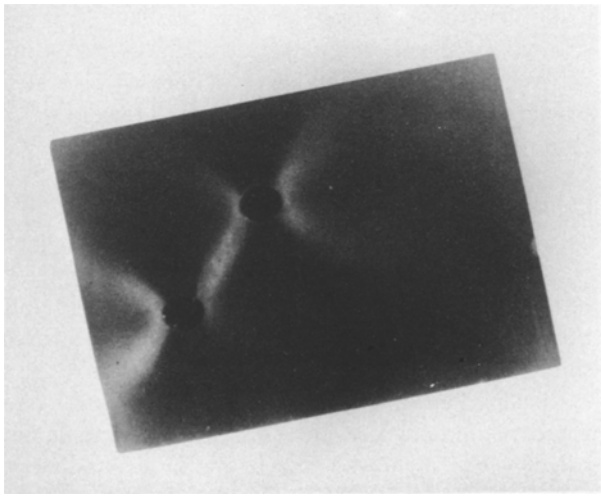

Figure 7 Stress whitening localized in bands around holes in a compression specimen tested at room temperature. Specimen diameter 18 mm.

With continued strain, the band becomes denser (whiter) and wider. In this investigation, compression tests were carried out to strains of 10% which generated enough damage to be observed at all temperatures and rates without the occurrence of fracture. All compression specimens were severely whitened, independent of temperature and rate of deformation.

A typical example of a stress-whitened compression specimen compared with an undeformed specimen is shown in Fig. 5. Barrelling is evident in the deformed specimens and some buckling occurred at the higher temperatures. When sectioned longitudinally, dead zones caused by the friction at the die–specimen interface are visible (Fig. 5c). The material inside the dead zones is relatively undeformed, being subject only to hydrostatic compression, and is thus undamaged. Virtually all the deformation and damage occurs between the two dead zones in the deformation zone.

Stress whitening in compression is visible regardless of temperature in the range investigated (23 to 105° C) for the silica-filled epoxy resin. Preliminary results indicate that stress whitening is more uniform and homogeneous throughout the specimen in creep compression than in short-time tests. Zones depleted of particles, i.e. pure resin zones, do not stress whiten. Failure in uniaxial compression occurs by the formation of a crack at the interface between dead zone and deformation zone as can be seen in Fig. 5d.

In order to observe the interior damage caused by compression, previously compressed specimens were impact-fractured perpendicular to the direction of uniaxial compression. Fig. 6a is an SEM micrograph of an undeformed specimen fractured in impact. In this case the fracture is matrix-dominated. The particles are undamaged and well bonded to the matrix. Fig. 6b is an SEM micrograph of a specimen deformed in uniaxial compression which was subsequently fractured through the stress-whitened damage zone, perpendicular to the compression direction. All visible particles are debonded and some are fractured. Thus two forms of damage resulting from compressive deformation have been identified: debonding and particle fracture, debonding being much more extensive than particle fracture.

As discussed previously, stress whitening is due to

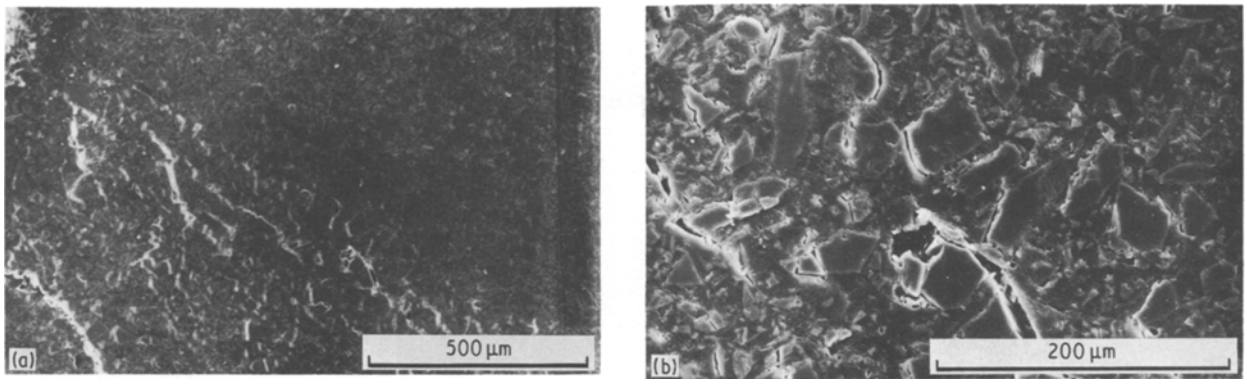

Figure 8 SEM micrographs of stress whitening between holes in a compression specimen, showing debonding. In some cases the matrix between the particles has failed; with further strain a network of microcracks will develop and coalesce into a propagating crack. The compression direction is vertical.

the presence of surfaces which scatter light within the solid. In order to observe stress whitening more carefully, holes were drilled in cylindrical specimens and the specimens were deformed in uniaxial compression, sectioned longitudinally, and then polished.

The resulting specimens whitened locally between the holes as shown in Fig. 7. Under higher magnification in the SEM (Figs 8a and b), the character of the damage can be observed in detail. Particles are debonded from the matrix on planes parallel to the shear stress and are the leading cause of stress whitening. Particle fracture is evident and occurs primarily on planes parallel to the compression direction. In addition, it can be seen that the matrix between debonded particles is beginning to fracture.

Stress whitening, as illustrated above, is primarily due to particle–matrix debonding with minor contributions due to particle fracture and matrix cracking. At higher strains in compression, microcracking between debonded particles contributes significantly to stress whitening as shown in Fig. 9. Eventually these microcracks coalesce resulting in failure, as shown in Figure 5d.

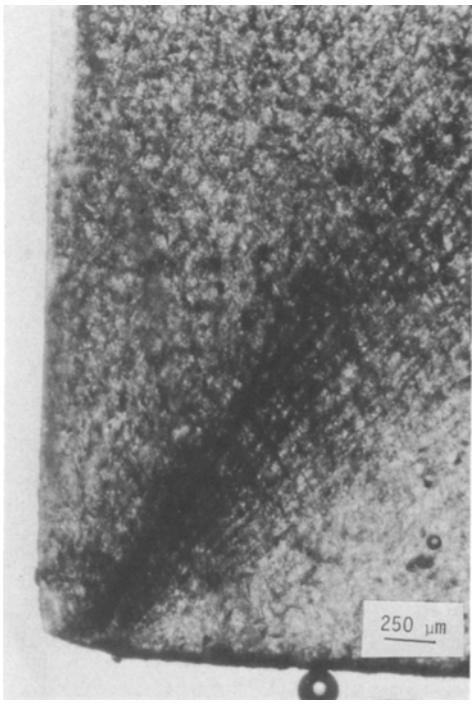

Figure 9 Transmitted-light micrograph of stress whitening in the corner of a compression specimen. Here a network of microcracks is evident.

In Section 3.1 it has been shown that pure epoxy resin exhibits shear yielding which may be localized by heterogeneities. Thus, it is expected that shear yielding occurs in the matrix surrounding the particles. Shear yielding in this material is best observed with polarized transmitted light under the optical microscope.

Figs 10a and b are examples of the undeformed and deformed material, respectively. In the undeformed material, the particles are undamaged and the matrix is uniform and undisturbed (Fig. 10a). Matrix shear yielding due to the compressive deformation is clearly evident in Fig. 10b. Highly birefringent shear bands permeate the matrix on planes parallel to the shear plane and, in addition, there is evidence of particle fracture. This photo is taken in a region of moderate stress whitening and thus only a few bands are evident. In regions of high deformation, the entire matrix volume is birefringent and it is impossible to distinguish individual bands.

In the highly filled material, the resolution of this technique is poor and deformation damage is often hidden due to the close proximity of the filler particles. Furthermore, it is difficult to see all types of damage in one photo with 42% filler as the polars must be crossed to observe shear bands, while particle debonding is best observed with unpolarized light. By lowering the volume fraction of filler in the material it is possible to observe all types of damage in a single micrograph and, in addition, it is easier to resolve individual shear bands.

Compression specimens with 16% (as opposed to 42%) volume fraction of filler were compressed uniaxially. The 16% material stress-whitens in the same manner as the 42% material. The whitening, however, is not as dense and higher strains are required for equivalent whitening. It also appears that higher strains are attainable prior to fracture.

The effects of compressive deformation on the low volume fraction material are shown in Fig. 11, which

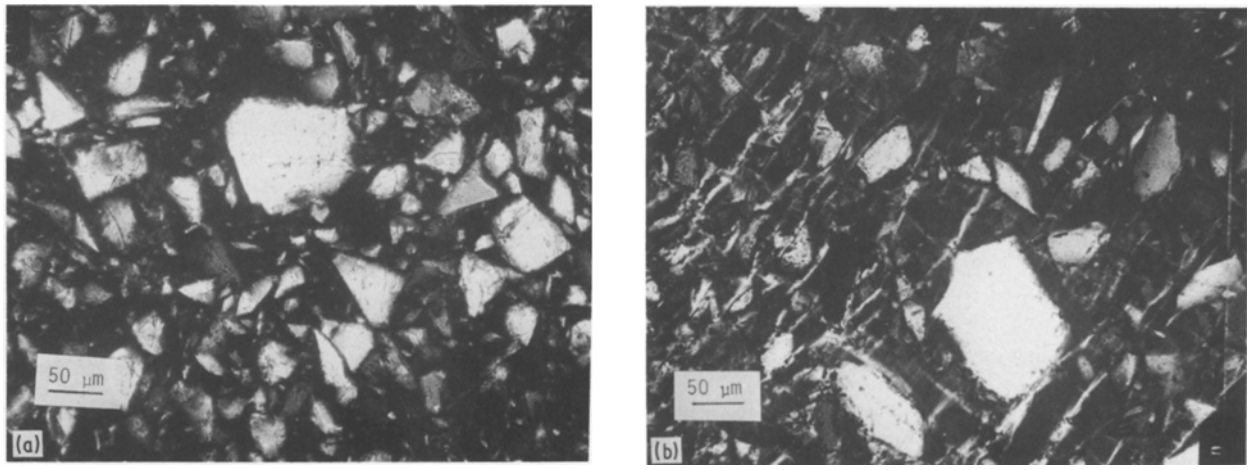

Figure 10 Transmitted polarized light micrographs of the 42% material. (a) Prior to deformation the particles are clearly evident and the matrix around them is undisturbed. (b) After compression the matrix is permeated with numerous shear bands; a fractured particle is also visible in the centre, but debonding cannot be detected without rotating the polars. Compression direction is vertical.

is a micrograph of the corner of a compression specimen. The compression direction is horizontal and the dead zone lies in the lower left. Debonding is evident as black regions around particles in this transmitted polarized light micrograph (Arrow 1). Fractured particles are also visible (Arrow 2). In the upper left corner (Arrow 3), debonded particles are linking up by matrix cracking. Finally individual shear bands (Arrow 4) are visible at the interface between the dead zone and the deformation zone. The shear yielding in the deformation zone (upper right) is so extensive that individual bands may not be resolved. The region is, however, highly birefringent.

It is observed that debonding in this 16% material is always accompanied by shear bands; however, shear bands are not always accompanied by debonded particles. In fact, shear bands appear to progress into the undeformed dead zones prior to any debonding (Arrow 4).

3.3. Initiation and recovery of volume damage

In order to determine the point at which damage is initiated, a number of compression tests were carried out and halted at various strains. The tests were performed on both the 16% and the 42% material at

Figure 11 Transmitted polarized light micrograph of the lower volume fraction material, showing the corner of a compression specimen. The compression direction is horizontal and the dead zone is in the bottom left half of the photograph. All types of deformation damage are visible in this micrograph and are indicated by arrows: (1) debonded particle, (2) fractured particle, (3) debonded particles linking up by matrix microcracking, (4) birefringent shear band.

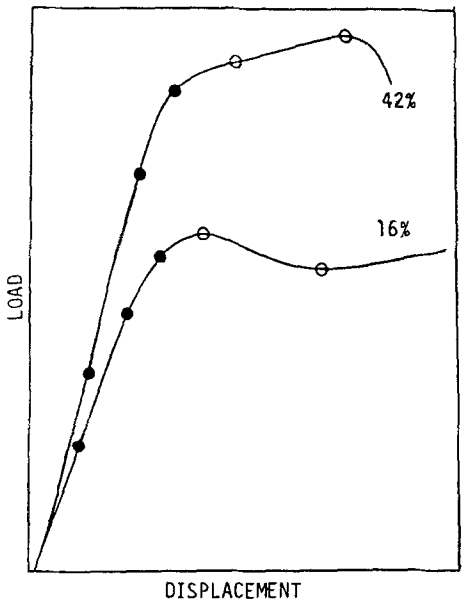

Figure 12 Schematic load-displacement curves for the normal (42%) material and a low volume fraction material (16%) in compression. The curves are typical of room-temperature tests. Each circle on the curves represent a point at which a test was stopped and a specimen was sectioned and examined for damage. (○) Stress-whitened samples, (●) specimens which exhibited no detectable internal damage or stress whitening.

room temperature. A typical load-displacement curve is shown schematically in Fig. 12. The solid points represent specimens which were not stress-whitened at that strain. The hollow points represent stress-whitened samples. The division between the two was relatively abrupt and could be observed with the naked eye. Subsequent microscopic examination revealed that shear yielding and debonding were only detectable in stress-whitened specimens.

It is well known that plastic deformation in highly crosslinked polymers is recoverable upon heating close to or above the glass transition [1]. It has been reported that stress whitening due to cavitation of rubber particles in elastomer-modified PVC is similarly recoverable [23]. It was thus of interest to determine the effect of heating on the silica-filled composite.

Specimens tested as described above were heat treated for 3 h at 130° C after deformation. They were then compared macro- and microscopically with specimens deformed to the same strain without post-heat treatment. All specimens showed greater than 99% recovery of strain after thermal relaxation. The stress whitening, on the other hand, was unaffected by the heat treatment. Microscopically, the extent of debonding was unaffected; however, all shear yielding detectable in polarized light vanished. This confirmed the supposition that stress whitening is not caused by shear banding in this material.

3.4. Tensile damage in filled resins

As a general rule, stress whitening is much less evident in tensile deformation. In tensile strength tests at room temperature, specimens with 42% filler volume fractions fail in a brittle manner at strains of 2% or less with no evidence of stress whitening. In creep-rupture tests, however, strains of up to 8% are attainable at higher temperatures (85 to 105° C) and longer times (more than one year) in the 42% material. These long-term high-temperature specimens stress-whiten severely prior to failure. The lower volume fraction material stress-whitens in strength tests above 85° C (at 0.5 mm min^{-1}). However, reductions in deformation temperature or increases in deformation rate suppress all visible macroscopic damage.

In short-term tensile strength tests with 42% material, there was evidence of microscopic volume damage only at the highest temperature (105° C) and slowest displacement rate (0.5 mm min^{-1}). Essentially, at lower temperatures, the first particle that debonds acts as a starter crack which instantly propagates through the gauge section resulting in failure.

In the same tests with the low volume fraction material, microscopic damage can be generated at the higher temperatures. Fig. 13 shows a transmitted polarized light micrograph of the area in the region of the fracture surface of a 16% filled sample tested at 85° C and 0.5 mm min^{-1}. Extensive shear yielding is evident in the region of the fracture surface. The

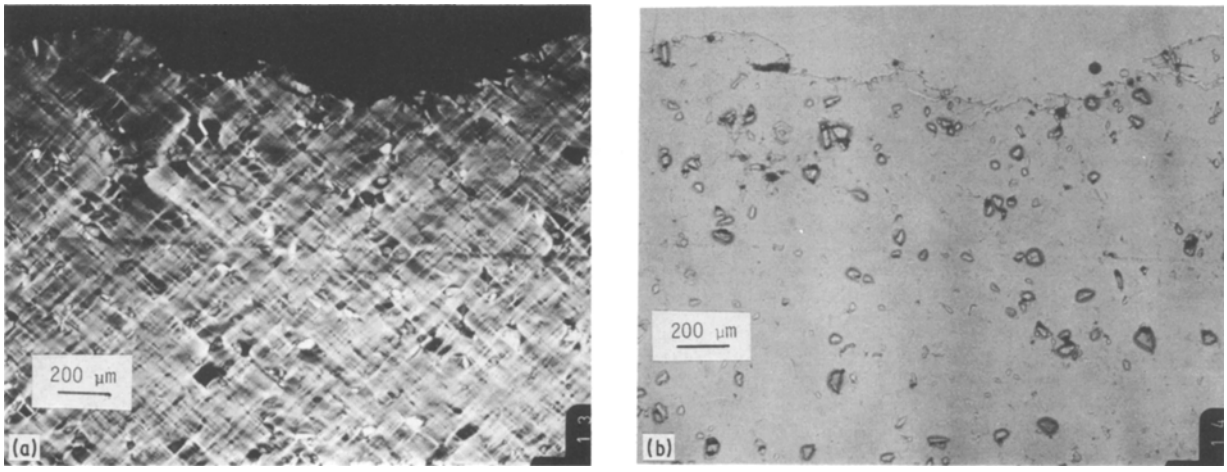

Figure 13 Transmitted-light micrographs of the 16% material deformed in tension at 85° C and 0.5 mm min^{-1}. The tensile direction is vertical and the fracture surface is visible at the top of each photo. (a) Polarized light micrograph showing extensive shear banding. (b) Rotating the polars extinguishes these bands, making debonding and particle fracture visible.

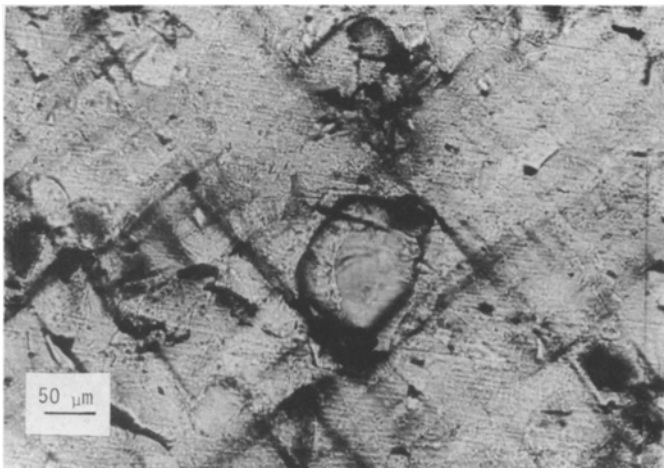

Figure 14 Polarized-light micrograph showing the interaction of shear banding and debonding around a particle in tension. The material is filled to 16% and the tensile direction is vertical.

specimen was slightly necked, and the region outside the neck showed only occasional shear bands. Rotating the polars on the microscope extinguishes the shear bands (Fig. 13b) and debonding is then visible as dark regions at the poles of particles. Fractured particles are also evident in the upper left quarter of the micrograph. Fig. 14 is a close-up of a particle; the matrix can be seen to be separated from the particle at both poles and shear bands radiate from these points.

The effect of increasing the crosshead speed by a factor of a hundred (0.5 to 50 mm min^{-1} at 85° C) is shown in Fig. 15. The numbers of shear bands as well as debonded particles are drastically reduced from those in Fig. 13 and macroscopic stress-whitening is absent. At lower temperatures there is no evidence of damage preceding brittle fracture.

In contrast to short-term strength tests, microscopic damage is evident in creep tests at 105° C in the 42% filled material, in the form of extensive debonding and some particle fracture (see Fig. 16). The tension direction is vertical and the debonding extends from the particle poles into the matrix, forming microcracks. These matrix microcracks can then link up with others, creating starter cracks as shown in Fig. 17. At 105° C and the longest times these microcracks contribute to stress whitening; however, the primary contribution is debonding. Only a few faint shear bands could be observed in these specimens.

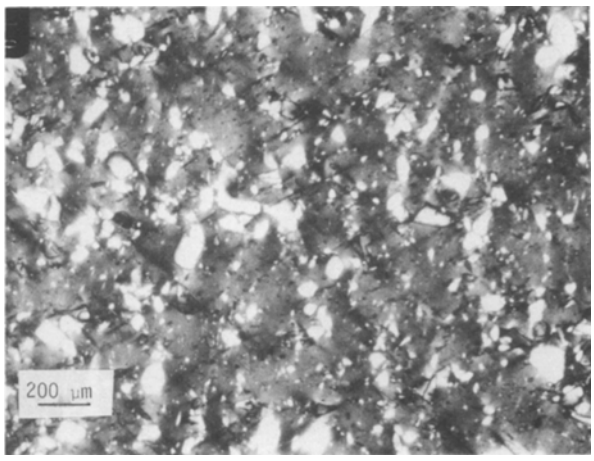

Figure 15 Polarized transmitted-light micrograph of the 16% material tested in tension at 85° C and 50 mm min^{-1}. When compared with Fig. 13a there is a significant reduction in the number of shear bands. In addition, although not visible in this micrograph, debonding and particle fracture are also significantly reduced.

4. Discussion

4.1. Pure resin

In spite of its generally brittle nature, the highly cross-linked unfilled epoxy resin can exhibit bulk shear yielding. Bulk shear yielding occurs at all temperatures (23 to 105° C) and all rates (0.5 to 5 mm min^{-1}) in compression and is possible in tension at higher temperatures. The epoxy resin is typical of most polymers [3, 18, 19, 24] in that yielding is enhanced by decreases in deformation rate and increases in deformation temperature.

In addition to bulk yielding, this epoxy resin has been shown to be very sensitive to defects or inhomogeneities. In the brittle case the heterogeneities initiate rapid failure. In the ductile case they promote localized shear yielding in the form of shear bands. These bands are highly birefringent and propagate at angles of between 45 and 50° to the direction of principal stress and are similar in character to those found in polystyrene [11, 12]. This shear yielding, localized or in bulk form, is completely recoverable upon heating for a few hours at temperatures above T_g as reported by others [1, 24, 25].

4.2. Filled resin

Adding silica particles to the epoxy matrix introduces inherent heterogeneities into the material which can promote extensive localized shear yielding around the particles. In addition, it has been shown that deformation in these particulate-filled epoxy resins can be almost completely recovered by heating for a time above the glass transition temperature. These results support the conclusions of Young *et al.* [3] who found that the incorporation of particles in epoxy did not change the fundamental deformation mechanism of the matrix, which is shear yielding. The incorporation of the filler does, however, introduce additional damage mechanisms including particle fracture and particle–matrix interfacial debonding, which at large strains leads to matrix microcracking.

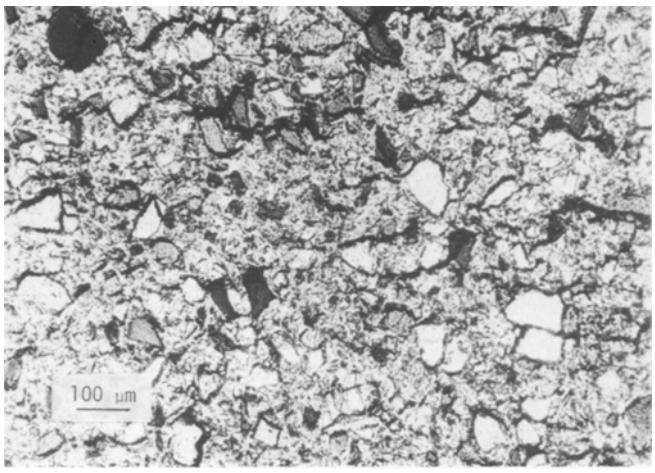

Figure 16 Polarized transmitted-light micrograph of a tensile creep specimen showing extensive debonding and particle fracture. Material is 42% tested at 85° C. The tensile direction is vertical.

The principal damage mechanism was found to be particle–matrix interfacial debonding. In contrast to shear yielding, which is a reversible deformation mechanism, debonding is an important irreversible damage mechanism. In tension, debonding occurs at the particle poles due to the inability of the particle–matrix interfacial bond to support the high dilatational stresses. The character of this damage is in agreement with both theoretical and numerical calculations [1, 14, 26] and also experimental observations [14, 17] reported in the literature.

In compression, debonding is always found to occur on particle surface planes oriented parallel to the shear plane. Debonding, in this case, appears to be due to the inability of the interfacial bond to accommodate the large shear deformation gradient between the particle and the matrix.

In our experiments, particle fracture plays a subordinate role. It might be more important when weaker particles such as aluminium hydroxide or dolomite are used [2], at higher filler loadings [18] or in the presence of better particle–matrix adhesion.

There was found to be no pronounced temperature or rate dependence of either debonding or shear yielding in compression. The extent of damage varied with the extent of total compressive strain. Furthermore, it has been shown that for all practical purposes damage initiates at the yield point. It is not possible to say definitely at this stage which occurs first, debonding or shear banding. However, we may tentatively suggest that matrix shear yielding occurs and gives rise to particle debonding. This, in turn, may enhance further matrix shear yielding.

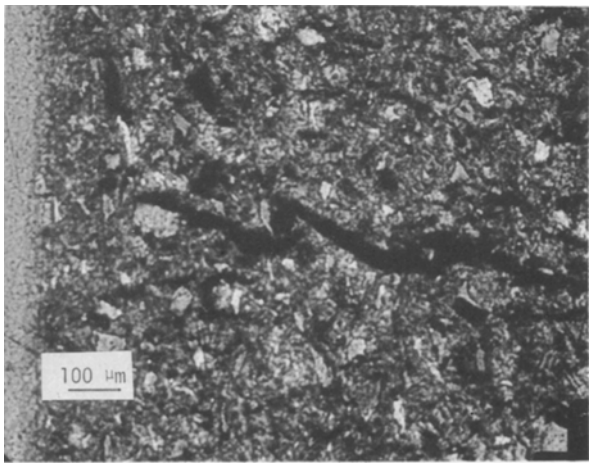

Figure 17 Polarized-light micrograph of crack formation due to the linking up of debonded zones in a tensile creep specimen (42% material) tested at 85° C. Tensile direction is vertical.

In tension, damage in filled resins could only be detected at 85° C, and above, for the 16% material, and only in creep at 85 and 105° C in the 42% material. This variation in behaviour with temperature and rate is not due to any change in manifestation of the mechanisms but rather it is due to the competition between ductile and brittle failure.

Macroscopically the ductility depends on the competition between shear yielding and fracture. At low temperatures and fast rates in tension the material is very sensitive to defects, and fracture occurs at the first occurrence of damage. Raising the temperature or lowering the rate increases the ductility and thus the notch sensitivity is reduced, permitting more damage accumulation. In compression, brittle fracture is suppressed so damage can accumulate freely.

5. Conclusions

1. The unfilled epoxy resin deforms by shear yielding and is very sensitive to the presence of inhomogeneities, which in tension at low temperature and high levels of load initiate brittle fracture in tension. In compression, and at higher temperatures and low load levels in tension, the inhomogeneities initiate shear banding.

2. The types of deformation-induced damage that have been identified in silica-filled epoxy resins are shear banding, particle debonding, particle fracture and matrix microcracking.

3. Shear bands form a network between particles at an angle of approximately 45° to the principal stress direction. They are highly birefringent, and may be completely recovered upon heating above T_g.

4. In compression, particle debonding occurs on particle surface planes approximately parallel to the shear plane. In tension, debonding occurs at the particle poles; that is, in planes perpendicular to the stress direction.

5. Particle fracture is much less extensive than

debonding and shear yielding. In compression, particles fracture parallel to the principal stress.

6. In compression, there is no pronounced time or temperature dependence of the accumulated damage preceding failure. In tension, brittle failure severely limits the damage accumulation and volume damage is only detectable at elevated temperatures and very slow rates, where the material is less sensitive to the stress concentrations produced by the damage.

7. Stress whitening starts at the yield point and represents irrecoverable damage in this material caused by cavitation. Particle debonding is the primary cause of this cavitation; however, at higher strains in compression matrix cracking will also contribute to stress whitening.

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