JOURNAL OF MATERIALS SCIENCE 10 (1975) 443–452

The influence of layer and bond strengths on the ductility of an all beryllium laminate

D. BEASLEY, J. E. J. BUNCE
Atomic Weapons Research Establishment, Aldermaston, Berks, UK

A limited study has been made of the effect of layer and bond strengths on the longitudinal tensile failure mode of an all beryllium laminate comprising a ductile (~ 5% elongation), high purity substrate and an adhering, brittle (< 1%), layer. It is proposed that the overall ductility is related to the extent of delamination at the interface which, in turn, is determined by the ratio (R) of bond strength to layer strength. When R is 0.23 extensive delamination along the gauge length is obtained resulting in maximum ductility, but at $R = 0.41$ little separation occurs and the elongation is reduced almost to the minimum, namely that of the brittle layer. A simple calculation indicates that for the material used in this work, the influence on ductility of this mode of failure in the brittle condition may begin to be reduced when the layer is < $\frac{1}{3}$ of the total composite thickness, when multipoint delamination may be anticipated.

1. Introduction

The behaviour of most composite materials is greatly influenced by the characteristics of the interfaces between the component phases. For example, in the most common application, where greater strength is achieved by the incorporation of strong fibres, the shear strength of the bond between them and the matrix must be sufficient to load the fibres fully. In a different context it has been proposed that catastrophic crack propagation could be prevented by delamination at an interface just ahead of a running crack. In this situation it has been shown by Cook and Gordon [1] that the ability of the interface to delaminate effectively is dependent on the ratio (R) of the strength of the matrix-second phase interface and the strength of the second phase. They calculated that if R < 0.2 to 0.3 delamination and hence crack arrest would occur, but if R exceeded these limits the crack would cross the interface and continue to propagate.

An opportunity to examine this hypothesis was presented by the availability of a limited quantity of a duplex, all beryllium laminate, consisting of a ductile (~ 5% elongation) substrate with an adhering brittle (< 1%) layer. A particular feature of this material is that the strength of both the brittle layer and of the bond respond to thermal treatment at temperatures too low to affect the mechanical properties of the substrate.

2. Material

Two nominally identical samples of material were used, designated A and B respectively, each comprising a substrate of HPEFP* grade beryllium consolidated by the CIP/HIP† route on top of which was formed an adherent less ductile layer of P10 (commercial purity) material. Sample A was tested in the as-fabricated condition but sample B was heat-treated for 24 h at 800°C in vacuum in order to increase the strength of the layer and its bond.

Specimens were cut in a common direction from each sample and tested at ambient temperature to evaluate the relevant parameters described below. In all tension tests the strain rate was 10^{-4} sec^{-1}.

3. Tests and results

3.1. Tensile properties of the substrate

Flat tension test pieces, of the design shown in Fig. 1, were etched after profiling to remove possible machining damage (0.05 mm per surface), and were subsequently tested to give the results shown in Table I. In this an asterisk distinguishes selected specimens from which

*HPEFP: High purity electrolytic flake powder.
†CIP/HIP: Cold isostatic pressing followed by hot isostatic pressing.

TABLE I The tensile properties of the substrate (all stresses in MN m^{-2})

Specimen number	L of P	Proof stress (%) 0.05	0.1	0.2	0.5	UTS	FS	El (%)	R of A (%)	Young's modulus ($\times 10^{-4}$)	Fracture code	Stress at F	El at F (%)
Sample A (as-fabricated)													
A2.1*	25	148	183	221	275					31.2	GE	315	1.2
A2.2*	27.5	152	188	223	275					30.2	GE	342	2.1
A2.3		196	215.5	239	268	362.5	376.5	4.0	3.7				
A2.4*	40	182.5	217.5	247	287	364	377	3.0	3.5	27.5	O		
A2.5		207	225	244	271	365	376	4.0	2.9				
Mean (A)	30.8	177.1	205.8	234.8	275.2	363.8	376.5	3.66	3.36				
Sample B (heat-treated)													
B2.1*	33	161.5	194	225	264	361.5	377.5	4.1	4.2	29.4	O		
B2.2*	30	155.5	185	214	253					25.7	GE	320	1.7
B2.3			192.5	216	253						GE	324	2.0
B2.4			204	225	256.5						GE	287	1.0
B2.5			192	216	252.5	361.5	374.5	4.0	3.5				
Mean (B)	31.5	158.5	189.5	219.5	258.5	361.5	376	4.05	3.85				

*Strain gauged.

Fracture code: O—failed (F) outside strain gauge, but within parallel gauge zone.
GE—failed near to grip end.

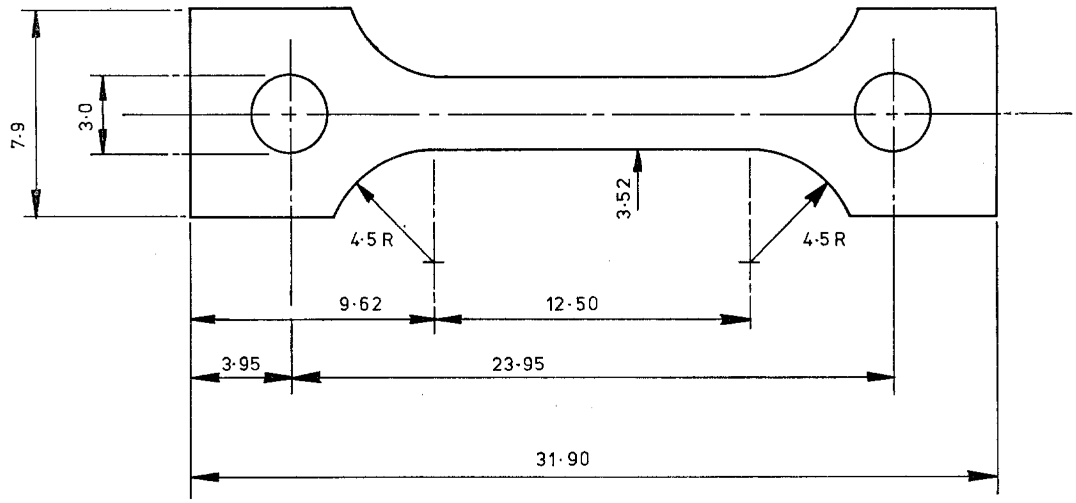

Figure 1 The tension test piece design. Dimensions in mm.

a precision stress-strain curve was obtained by combining and averaging the outputs from two 6.0 mm long strain gauges attached one to each of the opposite large faces of the gauge zone. The data from these tests (which were used to construct the stress/strain curves for the laminate specimens mentioned later) showed there was no significant difference in behaviour in the two conditions, thereby confirming the lack of response of the substrate to the thermal treatment.

3.2. Tensile properties of the layer

Material availability precluded the use of conventional test pieces and instead simple rectangular cross-section strips 2.5 mm × 1.5 mm × 55 mm long were employed from which only the ultimate strength could be obtained. The specimens, which were not etched, were pulled by cementing their ends into grips using Araldite. The results, given in Table II, show that the strength of the layer material was almost doubled by the heat-treatment. It has previously been established [2] that the ductility was < 1% in both conditions.

TABLE II Ultimate tensile strength of the layer material

Sample A (as-fabricated)		Sample B (heat-treated)	
Specimen number	UTS (MN m^{-2})	Specimen number	UTS (MN m^{-2})
A4.2	53.1	B4.1	111.5
A4.3	41.8	B4.2	18.3*
A4.4	25.4*	B4.3	92.4
A4.6	62.2	B4.4	80.0
		B4.5	7.0*
		B4.6	119.3
Mean (A)	52.3	Mean (B)	100.8

*These results have not been included in the mean. They are assumed to be premature failures attributable to machining damage (this series of specimens was not etched).

3.3. Bond strength measurements

The strength of the bond between the layer and substrate, see Table III, was determined by a test implemented by Calow and described in detail elsewhere [3]. This involved a straightforward tensile extension perpendicular to the interface, i.e. it was a "pull-off" test as distinct from the more widely used shear type. The bond in the B

TABLE III Substrate/layer bond strengths (pull off normal to interface)

Specimen number	Fracture stress (MN m^{-2}) Sample A (as-fabricated)	Sample B (heat-treated)
1	10.33	4 tests in the
2	13.54	range 36.5 to
3	3.44*	45.7
Mean	11.98	41.1
Bond strength/layer strength ratio (R)		
Mean values	0.23	0.41

TABLE IV Longitudinal tensile properties of the duplex specimens

Specimen number	Thickness (mm)		% plastic strain in substrate at layer failure	(i) Estimate of stress in substrate at layer failure ($MN\ m^{-2}$)	(ii) Stress in substrate after layer failure ($MN\ m^{-2}$)	UTS for duplex, ($MN\ m^{-2}$)	FS of substrate, ($MN\ m^{-2}$)	El (%)
	Substrate	Layer						
Sample A (as-fabricated)								
A2.6*	1.93	0.5	0.17	240	258	281.5	363.5	2.9
A2.7	1.97	0.48	0.64	288	288	285	364	4.6
A2.8	1.94	0.48	0.48	277	287	286	367	4.2
Sample B (heat-treated)								
B2.6*	1.14	0.54	0.17	217	326	213	316	0.72
B2.7	1.20	0.46	0.26	235	306	225	311	0.96
B.28	1.05	0.62	0.07	172	320.5	200.5	320.5	0.40

*Specimen strain gauged, one on each face.

(i) This value was obtained from the stress/strain curves for specimens 2.1, 2.2, 2.3, 2.4 and 2.5 (from Table II).

(ii) This stress is the one to which the substrate is instantaneously loaded when the layer fails. It was calculated by taking the load at the point of layer failure and dividing by the substrate cross-sectional area.

material (heat-treated) was 3 to 4 times stronger than that of the A samples and these data combined with those for the layer strengths (Table II) yield values for R (the ratio of bond to layer strengths) of 0.23 (A) and 0.41 (B) which conveniently span the Cook and Gordon threshold (< 0.2 to 0.3).

3.4. Tensile properties of the laminate

Duplex tensile specimens (Fig. 1) were cut with their thickness astride the substrate/layer interface such that they comprised a strip of each component; they were etched before testing. Selected specimens were strain gauged on each face (i.e. substrate and layer), but in this case the outputs were individually recorded in order to monitor simultaneously the individual behaviour of the layer and the substrate with respect to load up to the point of their respective failures. The results of all the tests are summarized in Table IV which also includes the stresses in the substrate, (a) at the instant of failure of the layer, (derived from the strain indicated by the gauge, and the known characteristics of the substrate), and (b) immediately after failure of the layer, calculated by assuming that the total load was instantaneously transferred to the substrate. Using these latter data, together with the load-strain values measured after layer failure, and making the assumption that the properties of the substrate were not significantly affected by the layer, complete stress-strain curves for the substrate portions of the duplex specimens have been generated. These are presented in Fig. 2 together, for comparison, with curves for the monolithic substrate specimens.

During the testing of the sample A specimens, the initial crack in the layer was audible as a distinct click, following which it slowly delaminated from the substrate in both directions along the gauge length as the strain in the substrate was increased; in one instance pieces were completely detached. This behaviour was associated with overall ductilities very similar to those for the monolithic substrate. By contrast, the sample B specimens failed almost immediately, (specimen B2.8), or quite shortly after the first crack in the layer, (specimens B2.6 and B2.7),

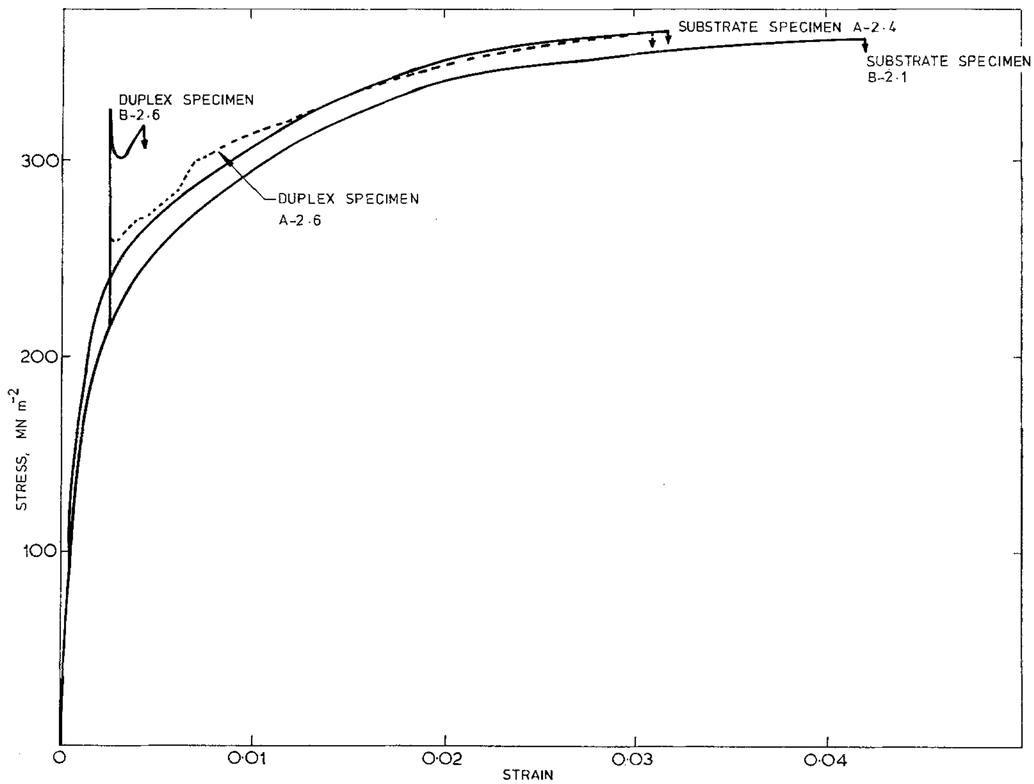

Figure 2 Comparison of the generated stress/strain curve for the substrate portion of a duplex specimen with stress/strain curve for a monolithic substrate specimen; examples from both A and B are shown.

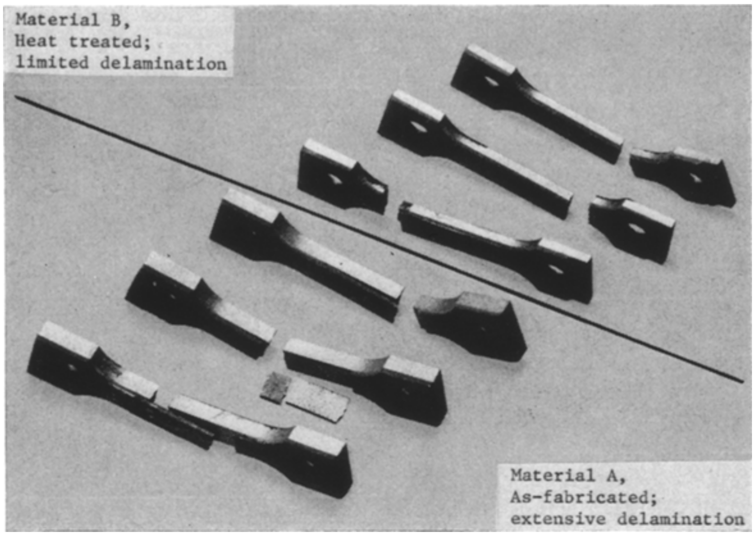

Figure 3 Appearance of laminate test pieces after fracture, $\times \sim 1.25$.

without any obvious delamination and at elongations up to an order of magnitude less than those of sample A. The appearances of the broken test pieces are compared in Fig. 3, whilst Fig. 4 shows the fracture of one specimen from both samples at low magnification.

3.5. Metallographic examination of the laminate specimens

Longitudinal sections from the axes of the specimens were examined metallographically, particular attention being given to the interface and fracture regions, see Fig. 5. In addition, their fracture surfaces were viewed in the scanning electron microscope, some of the major observations from which are shown in Fig. 6.

The extensive delamination characteristic of the sample A (as-fabricated) material occurred mainly at the interface in specimen A2.8, with localized deviations into the layer in A2.7 and with appreciable failure in the layer in A2.6, suggesting that the strength of the bond was comparable with that of the layer itself. Although the fracture of the layer instantaneously increased

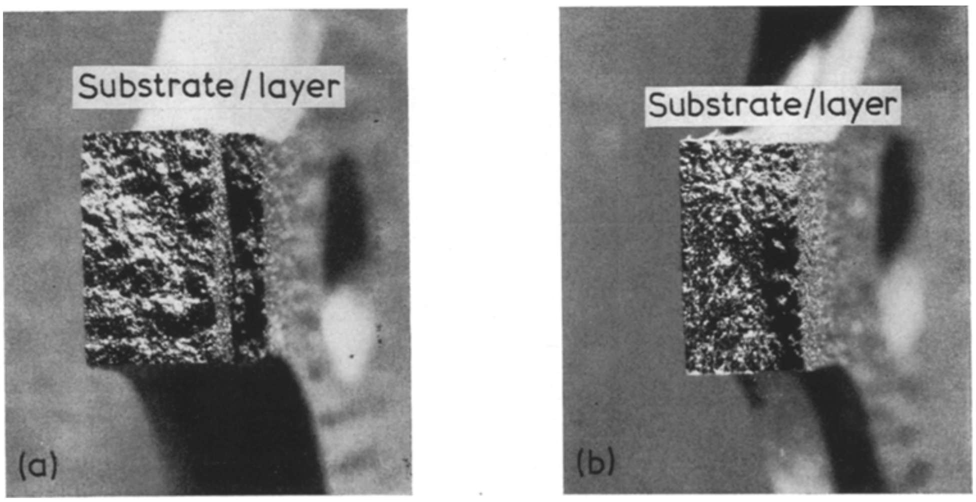

Figure 4 A low magnification fractograph of two broken laminate tensile specimens. (a) Specimen A-2.8, as-fabricated condition. (b) Specimen B-2.8, heat-treated condition. $\times \sim 10$.

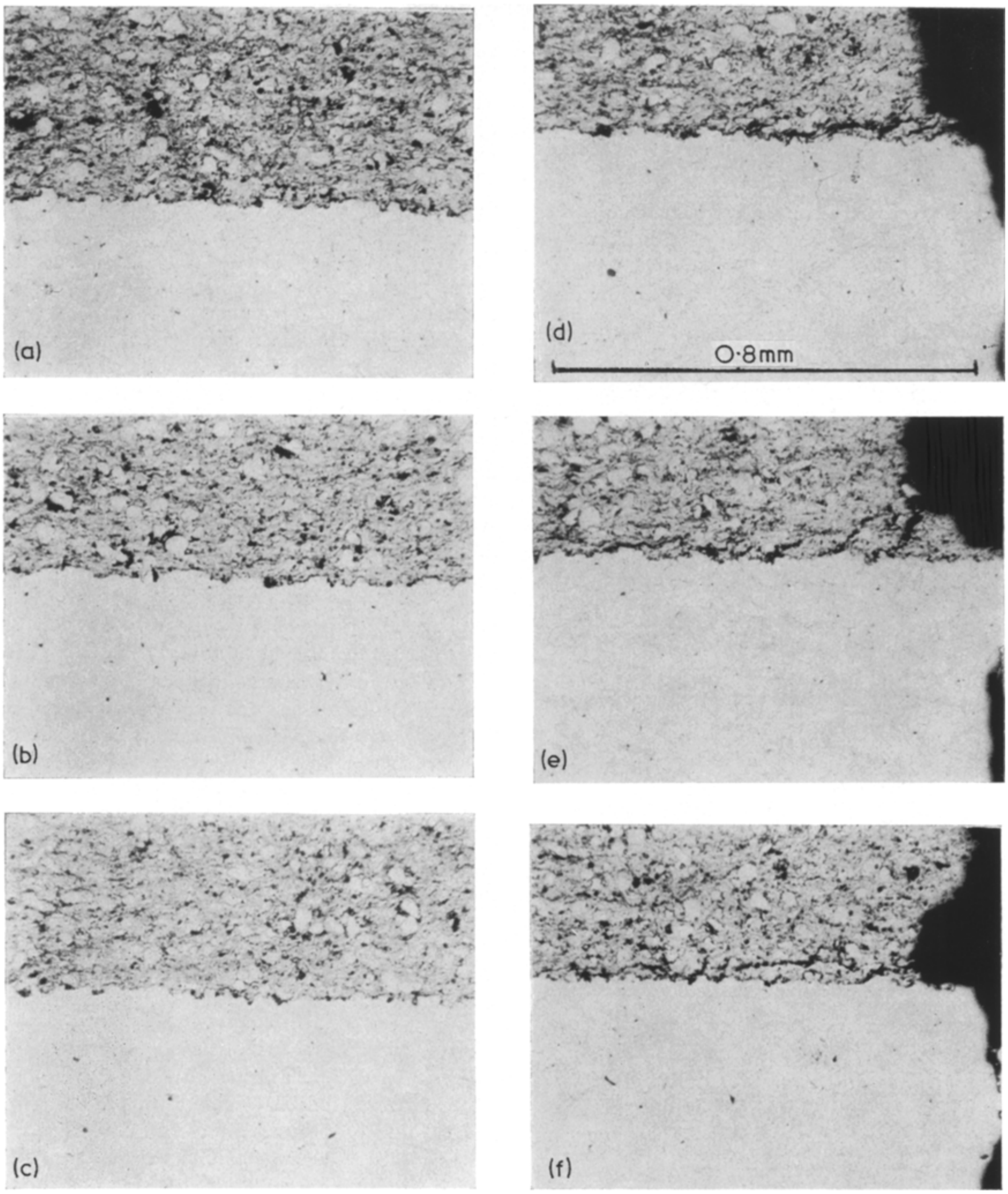

Figure 5 Longitudinal sections at and near the fractured ends of the heat-treated (sample B) laminate specimens: (a) to (c) 1 to 2 mm back from the fractured ends; (d) to (f) fractured ends. (a) and (d) specimen 2.6, (b) and (e) specimen 2.7, (c) and (f) specimen 2.8.

the stress in the substrate by $\sim 10\%$, see Fig. 2, this did not cause the latter to fail and the tests continued to final fracture with the substrate exhibiting its mono-lithic properties. The extensive delamination served, therefore, to isolate quite effectively the two components.

However, in the heat-treated condition there was no such isolation, the microscopic examination (Fig. 5) showing that delamination was confined to a narrow zone adjacent to the layer

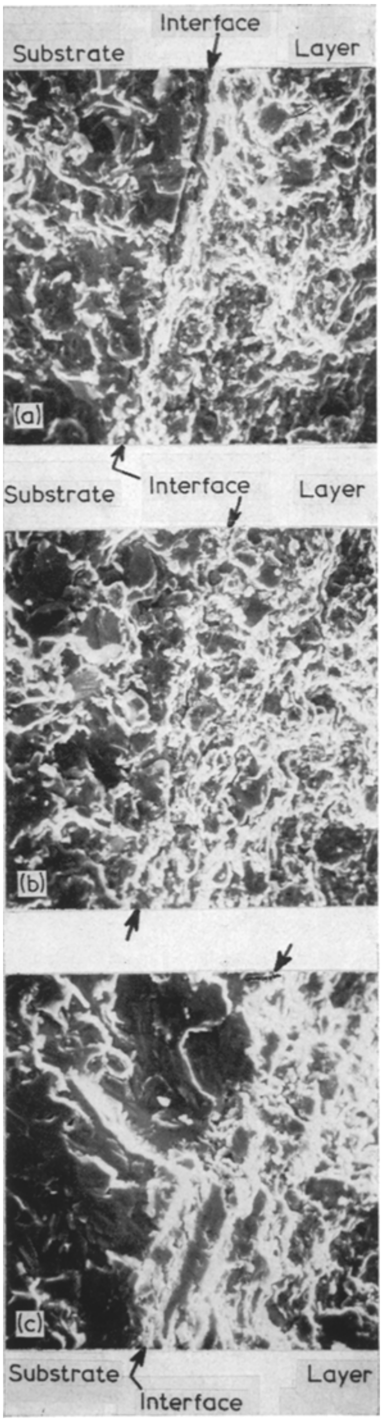

Figure 6 SEMs of the fractured ends of the heat-treated laminate specimens (sample B). Specimens (a) 2.6, (b) 2.7, (c) 2.8, × 175.

(and specimen) fracture beyond which the interface was intact with the layer firmly adhering to the substrate. In specimens B2.6 and B2.7 this zone extended ~ 0.8 mm along the gauge length in both directions, corresponding to a total delaminated "gauge length" of ~ 1.6 mm, whilst in specimen B2.8, which failed almost immediately, the zone was only ~ 0.4 mm long.

4. Discussion

4.1. General

The difference in behaviour of the laminate specimens in the two conditions (A and B) is in basic qualitative agreement with the concept proposed by Cook and Gordon. Although, strictly, delamination would not have been predicted for B ($R = 0.41$, > 0.3) the principle of reduced delamination with increase in the ratio is clearly evident. In both conditions, therefore, delamination occurred to prevent catastrophic crack propagation from the layer through the substrate, but, regardless of this, the overall ductility was very markedly reduced by the heat-treatment.

4.2. Proposed explanation for the effect of heat-treatment on ductility

Although premature fracture could have been induced by the shock loading when the layer broke, the steady increase of load after layer failure observed in two of the three heat-treated specimens indicates this is not the case. Instead we propose that the overall elongation is decreased because the effective gauge length is reduced to that length of substrate from which the coat has become delaminated, i.e. for the present work ~ 1.6 mm.

This is supported by a simple calculation for the test on specimen B2.6. In this the elongation to fracture for the substrate measured over the whole gauge length (0.72%) was double that measured on the portion covered by the strain gauge (0.36%) which did not include the region of the fracture. If it is assumed that the strain gauge value was representative of the strain in the substrate over the portion of the gauge length where the bond was intact, the elongation of the substrate which occurred in the reduced, (delaminated), gauge length can be obtained from the equation:

$$L\epsilon = (L - l)\epsilon_s + l\epsilon_g$$

in which L = overall gauge length, ϵ = overall fractional elongation, l = estimated length of delaminated interface, ϵ_s = fractional elongation measured by strain gauge on substrate,

ϵ_g = fractional elongation on reduced gauge length.

For specimen B2.8 which failed quickly the values of ϵ, ϵ_s and ϵ_g were effectively all the same, but for B2.6 and B2.7 using the measured delaminated length of 1.6 mm yields values for ϵ_g of 3.2 and 4.5% respectively which agree well with the properties for the (monolithic) substrate specimens.

It follows from this hypothesis that the overall elongation of duplex specimens, comprising a less ductile layer on a stronger, more ductile substrate, is related to the bond/layer strength ratio since this determines the extent of delamination. The range of overall ductility obtained will extend from that of the most ductile component, achieved when there is extensive delamination, down to that of the less ductile layer when there is no delamination at all to arrest the crack. It should be noted that when limited delamination occurs, the observed overall ductility will vary with the length of the specimen since the latter will determine the percentage contribution of the delaminated region.

4.3. The effect of layer thickness

Although, fortuitously, the specimens in the present work had various layer/substrate thickness ratios, there are not sufficient data to enable the practically important relationship between this parameter and overall ductility to be established; however, it is possible to make a limited hypothetical prognosis. For the two extreme conditions of layer behaviour, namely complete delamination and no delamination, it is probable that the overall ductility will be independent of the layer thickness, since the substrate and layer characteristics, respectively, will predominate. For the intermediate condition in which limited delamination occurs (as seen for the heat-treated material), the overall ductility is determined by the extent of delamination and will, therefore, be largely independent of layer thickness. However, this behaviour will be changed when the layer is sufficiently thin with respect to the substrate for work hardening in the delaminated region to reload the remainder of the specimen to such an extent that the layer cracks and delaminates again elsewhere. This new delaminated region, which will not have work hardened as much as the initial one, will then become the focus for deformation and the process could be repeated. Successive repetitions may be obtained, each one providing an additional increment to the total elongation such that under the most favourable conditions the full ductility of the substrate may possibly be realized. An indication of the critical layer/composite thickness ratio for this behaviour may be obtained using the data in Table II. Thus, at the point at which the layer, (thickness t), fails the stress in the specimen is 220 MN m^{-2} which, for a specimen of total thickness T, corresponds to a load of $220T$ MN. This must then be sustained by the substrate alone, (ultimate strength 360 MN m^{-2}), from which it follows that;

$$\frac{T-t}{T} \geqslant \frac{220}{360}$$

and hence $t \leqslant T/3$. Thus, for possible multiple cracking the layer must be less than a third of the total specimen thickness.

4.4. An appraisal of the significance of the reduced ductility to structural applications

It is to be expected that failure of this form of duplex material in a straight tension test will be initiated in the layer since this has the lower ductility (and strength). The mode of failure of the composite will, therefore, depend on the way this initial crack propagates; it may:

(a) accelerate through the interface and, by virtue of the stress concentration at the crack tip and the high local strain rate, cause the normally ductile substrate to fail in a brittle manner;

(b) penetrate the interface but be decelerated and possibly even stopped in the substrate by local deformation at the crack tip;

(c) be arrested at the interface.

(d) be arrested at the interface which may delaminate.

Of these four, only the first, (a), is really dangerous, representing as it does an all brittle situation which renders the composite susceptible to catastrophic failure. Moreover, it would eliminate the benefits of the duplex structure because failure would be determined entirely by the properties of the layer. Behaviour of this kind was not observed in the present work in which even specimen 2.8 from sample B, which fractured very shortly after the layer failed, appeared fractographically to have broken with some localized ductility (Fig. 6). The remainder, (b) to (d), are all situations in which catastrophic failure will be prevented and the structure safeguarded against premature failure. However, on the basis of the present model, complete delamination, e.g. (d), is required to realize the full potential ductility of the substrate.

5. Conclusions

(1) The quasi-static tensile properties of a duplex all beryllium composite comprising two adherent layers, one appreciably more ductile and stronger than the other, can be significantly changed by thermal treatment. In the example chosen here, heat-treatment for 24 h at 800°C, reduced the composite ductility by an order of magnitude from ~ 5.0 to 0.5%.

(2) A model is proposed for this phenomenon based on the observation that the stronger component was unaffected by the anneal. This relates the composite ductility to the ratio of the strength of the bond and of the less ductile layer (both of which were increased by heat-treatment), which in turn determines the extent of delamination at the interface ahead of a crack in the weaker layer as it propagates towards the stronger component.

(3) It follows from the model that the overall composite ductility may range from that of the most ductile component on its own (5%), associated with extensive delamination, down to that of the less ductile component (~ 0.5%) when there is little or no delamination and cracks can propagate through the interface into the more ductile region. Although not observed in this work, it is suggested that in the extreme, the composite could behave in a brittle fashion.

(4) On this basis a strong bond between the layers will inevitably reduce the composite ductility. However, the present work indicates that a bond/layer strength ratio of 0.23 is small enough to ensure complete delamination, and 0.41 is sufficient to prevent catastrophic brittle behaviour.

(5) It is emphasized that for structural applications the reduction of ductility due to thermal treatment may not be significant provided there is sufficient delamination just to prevent brittle (catastrophic) failure.

Acknowledgements

The authors would like to thank Dr C. A. Calow for the use of his bond and layer strength data, and Miss H. M. Lindsay for the Scanning Electron Microscopy.

References

1. J. COOK and J. E. GORDON, *Phil. Mag.* **282** (A) (1964) 508.
2. C. A. CALOW, AWRE, Aldermaston, private communication.
3. C. A. CALOW, AWRE, Aldermaston. To be published.

Received 29 August and accepted 9 September 1974.