

Effect of temperature and superimposed dynamic and static stresses on mechanical properties of epoxy-bonded joints

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Adhesive joints are subjected during their service life to different combinations of dynamic and static stresses. While the behaviour of the adhesives in relatively simple states of stress is well characterized, their response to superimposed stresses of different types acting in different directions has scarcely been investigated. In the study presented in this paper, single lap joints made with different formulations of epoxy adhesives were subjected to combined shear creep stresses and torsional oscillations applied simultaneously and along perpendicular axes of the specimen. The main conclusion based on the results of this investigation is that such a simple combination of stresses affects considerably the mechanical behaviour of the joint. A significant increase of the shear strength of the joint was recorded for specimens subjected to superimposed stresses at temperatures lower than the T_g of the adhesive. Application of similar combinations of stresses at temperatures close to or higher than the T_g led to a decrease of the shear strength of the joint. The fracture morphology of the joints made with the investigated epoxy resins was qualitatively correlated with changes induced by the superimposed loading in the T_g of the adhesive. The fatigue fracture surface of adhesives is characterized by striations and furrows, similar to bulk specimens that failed in the same fashion.

1. Introduction

The use of epoxy resins as adhesives in the aircraft industry has a history of more than 40 years [1] and is continuously expanding also in other fields. Consequently, efforts are being made in order to achieve a better understanding of the structure-property relationships of these resins in their bulk form, as well as adhesives in bonded joint systems.

It is well known that joints are subjected during their service life to a very complex system of stresses. A typical example is that of the joints in a car or an aircraft: while bearing the weight of the joined part (static stress), the adhesive is subjected to vibrations (dynamic stresses) whose amplitude and frequency depend on many factors related to travelling conditions. Therefore, characterization of joints using either monotonic or dynamic loading tests while disregarding the effect of other superimposed stresses may not serve, even qualitatively, for reliable predictions of joint performance under real service conditions [6].

Most publications which deal with this issue describe the effect of static [2, 3, 4] or cyclic stresses on adhesives [5–10]. The main effect encountered when cyclic stresses alone were applied on bulk resins and/or on joints, was a considerable reduction of their ultimate strength, when compared with specimens subjected to static stresses. The commonly accepted explanation for this phenomenon was based on the cyclic softening approach [6, 11, 12].

The fracture of epoxy resins in bulk and in adhesive joints has been intensely investigated, but the mechanisms which govern the process are still the subject of controversy [13–28]. Disagreement exists even about the question of similarity in fracture behaviour of the two forms of the same resin. Dolev and Ishai [29] and Gledhill and Kinloch [30] found that the basic mechanical behaviour is the same for both forms. They based this conclusion on comparative estimation of stress–strain curves and fracture morphology in both states, respectively. A different conclusion was reached by Mostovoy and Ripling [31] after considering the fracture toughness of amine-cured epoxides; according to them it is not possible to predict joint properties from the known behaviour of the adhesive in bulk. They found that the bulk fracture toughness of the resin is not always higher than that of the joint; there are cases in which, after high-temperature post-curing of certain resin-to-hardener ratios, the polymer in the joint is tougher than the same polymer in bulk.

To the best of our knowledge, there are no systematic investigations reported in the literature on the effect of simultaneously applied static and dynamic stresses on the mechanical properties of joints and there is no information on the fracture surface morphology of joints exposed to such loading conditions. There are a few studies done on bulk epoxy specimens (Lifshitz and Rotem [32] and Katz, Smooha and Isayev [12, 33]) subjected to torsional and elonga-

tional cyclic loading at different amplitudes, loads and temperatures, but they were limited to brief periods of time (from few seconds up to 1 hour).

The main goal of this paper is to obtain some insight into the effect of a simple combination of dynamic and static stresses on a model joint, to evaluate the importance of this effect and to correlate, at least qualitatively, changes occurring in the joint properties with changes in the microstructure of the adhesive.

2. Experimental procedure

2.1. Instrumentation

A special apparatus [34] was designed and built in order to apply a simple combination of dynamic and static stresses to single lap adhesive joints (Fig. 1). Shear creep was applied vertically to groups of five joints by means of weights pulling a rod (9) through a precise antitorion linear bearing (10). Torsional oscillations were applied horizontally (5) to the upper grip holding the specimen (1) using a variable eccentric (4) rotated by an electronically controlled (3) direct current (d.c.) motor (2). We believe that this combination may represent to some extent the real system of stresses acting on joints. The changing parameters were creep load, amplitude and frequency of torsional oscillations, the adhesive's composition, and temperature. The last parameter was maintained with a precision of $\pm 1^\circ\text{C}$ in an oven designed for this purpose and controlled with an Eurotherm regulator (11).

The experiments were carried out in ambient atmosphere for 24 hours except those interrupted by the specimen's failure (fatigue). Most of the specimens did not fail during loading and were subsequently tested in shear by tensile loading.

2.2. Specimens

The single lap type joint (SLJ) was chosen in order to duplicate the geometry and service conditions for many structural adhesives [35]. The main problem

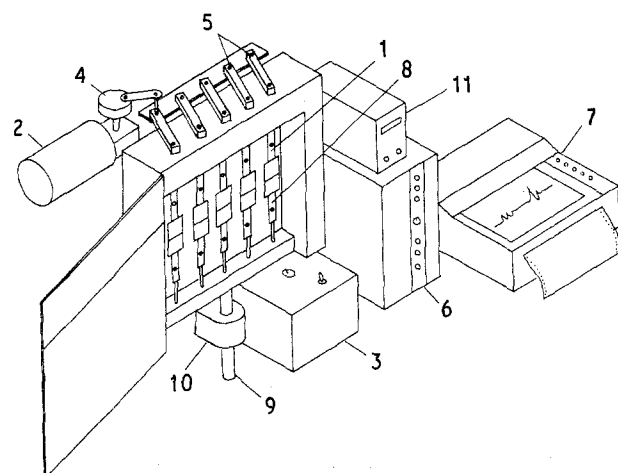


Figure 1 Apparatus for application of superimposed stresses; 1 - upper jaw-grips, 2 - d.c. motor, 3 - electronic r.p.m. controller, 4 - eccentric, 5 - upper arms, 6 - strain-gauge amplifier, 7 - recorder, 8 - lower jaw-grips, 9 - pulling rod, 10 - torsion-resistant linear bearing, 11 - temperature controller.

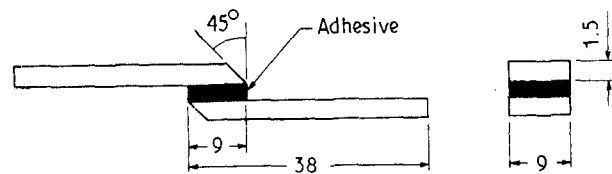


Figure 2 Schematic drawing of the single lap joint.

encountered with this type of joint is the non-uniform distribution of stresses, i.e. the maximum stress at the bondline may differ considerably from the average. Since the work does not deal with stress analysis within the joint, but mainly examines the influence of different working conditions on joint properties, this type of joint was used only for comparison, because we were aware that the non-uniformity in stress distribution may express itself in a relatively high standard deviation of experimental data, which was indeed the case. In order to minimize the stress concentration at the edge of the overlap area and consequently the standard deviation of strength data, a tapered modification of SLJ was used in this work [36-38]. The substrate material was aluminium 2024-T3 which was chrome anodized after being cut to size. The dimensions of the joints are shown in Fig. 2. The joints were prepared and cured in a specially designed device which assured a good three-dimensional alignment of the specimens [34].

The pressure applied on the bonded region during cure was constant and equal for all five joints. This was achieved by means of a calibrated spring and a plate running parallel on two columns attached to the base plate. As a result, the thickness of the adhesive layer was uniform over the whole overlap area and almost the same for all investigated joints.

2.3. Materials

The adhesives used in this work were based on Epon Resin 828 (made by Shell Co.). Different types of curing agents were used in order to obtain a large range of T_g values of the adhesives (Table I). With triethylene tetramine (TETA) a resin with T_g of about 117°C was obtained, while a combination of Epon Resin 828 and Thiokol LP-3 (a product of Thiokol Co.) yielded, with the same curing agent, a resin with T_g of about 55°C . By curing the Epon Resin 828 with a suitable proportion of Versamid 140 (manufactured by General Mills Inc.), an adhesive with T_g of about 6°C was obtained. All the relevant data are shown in Table I.

Since the superimposed stresses were applied in the temperature range $25-105^\circ\text{C}$, resins with different T_g allowed us to investigate the effect of loading on adhesives in their glassy, transition and rubbery regions.

The joint preparation began by cleaning the aluminium adherends with trichloroethylene and isopropylalcohol (30 min each). The adhesive components were mixed manually in ratios shown in Table I at room temperature and left for 30 min at the same temperature in order to allow the escape of air bubbles

TABLE I Adhesives composition (wt %) and curing conditions

	EV-1	EV-2	EV-3	E	ET
Epon Resin 828 (Shell Co.)	40%	50%	60%	90.91%	83.3%
Versamid 140 (General Mills)	60%	50%	40%	—	—
Thiokol LP-3 (Thiokol Co.)	—	—	—	—	8.33%
TETA (Fluka A.G.)	—	—	—	9.09%	8.33%
Room temperature curing (min)		30		30	30
Oven temperature (°C)		65		140	105
Oven curing time (hours)		3		3	0.3
Room temp. postcuring (days)		—		—	7
Glass transition temp. (°C)	5.8	52	56.2	105	39

introduced during mixing. Curing was performed in an oven with forced air circulation at temperatures and for periods shown in Table I. After curing, the joints remained in the oven for slow cooling during 24 hours. The absence of exothermal peaks in differential scanning calorimetry (DSC) measurements of the cured adhesives was accepted as a proof for completion of the curing reaction [39, 40].

2.4. Investigation techniques

2.4.1. Mechanical testing

A Zwick 1445 machine equipped with a load cell of 10000 N was used. The shear tests in tension were always conducted at room temperature and at a 2 mm min^{-1} crosshead speed. The mechanical testing of joints usually yields very scattered data: Grimes [8] reports experimental scatter of about 12–14% of the average in shear testing of single lap joints made with aluminium adherends and an epoxy adhesive; Arrow-smith and Maddison [41] report results with deviations from the average of up to 55% when working in humid conditions; the results revealed by Ikegami *et al.* [2] also have very high deviations from the average. Kurzman and Klemme showed that the strength of adhesive single lap joints obeys the Kase distribution law [42] and that the results follow an asymmetrical (negatively skewed) dispersion instead of a Gaussian one. A good approximation to the mode of the Kase distribution (i.e., the maximum of the distribution curve) can be obtained by averaging the highest three values from a five-specimen sample [42]. The same approach was adopted in this work after verification that it does not affect the physical meaning of the data (no other trends were found by processing the data differently).

2.4.2. Thermal testing

Some thermal properties of the adhesive were studied by means of DSC using a Mettler TA3000 DSC instrument. The weight of samples was about 10 mg and the heating rate was 10 °C min^{-1} . The main values obtained from DSC thermograms were T_g (at the intersection of regression lines of glassy and transition regions) and the transition zone width defined as the difference between T_g and T_{rubbery} (the intersection of regression lines of equilibrium and transition regions) [43].

The experimental error in T_g determination by DSC was $\pm 0.6 \text{ °C}$ (based on five identical samples) [44].

2.4.3. Scanning electron microscopy (SEM)

The studies of fracture surfaces were performed mainly on specimens for which the adhesive EV-2 was used for joint preparation. The overlapping areas ($9 \times 9 \text{ mm}$) (Fig. 2) of the specimens which failed during the course of the experiment were cut off from the remaining aluminium plates and bonded to cylindrical supports made of brass. The gaps were filled with conductive paint and the whole assembly was gold-coated in vacuum to a thickness of about 50 nm. The specimens so prepared were studied by means of a Jeol T-200 electron microscope at an acceleration voltage of 25 kV. By using a tilt angle from 0° up to 75° it was possible to investigate the fracture surfaces, both normal and parallel to the substrate. No special problems connected with specimen charging were encountered, but at high magnifications the specimens were damaged by the electron beam. This type of damage is relatively easy to identify: it appears as continuously increasing cracks at the centre of the investigated area and it can be avoided if the alignment of the microscope for exposure is done within 5–10 sec.

3. Experimental results and discussion

3.1. Mechanical properties

Fig. 3 depicts the effect of the Epoxy–Versamid 140 ratio in the adhesive on the average strength in shear of the tested SLJs. A rather mild combination of stresses was applied on the joints for 24 hours at room temperature: torsional oscillations with a frequency of 1 Hz and a creep load of 0.8 kg (which is roughly only 0.5% of the load at break of the joints). On the same graph are plotted the data for shear strengths of joints subjected and not subjected prior to testing to superimposed stresses. It can be seen that the shear strength of the joints depends not only on the adhesives' compositions, but also on their history. The decrease of about 55% of the shear strength of joints prepared with the composition EV-1 seemed predictable, according to the cyclic softening approach. An increase of about 33% in shear strength of the joints made with the resin containing 50% Epon Resin 828 and 50% Versamid 140 was somewhat surprising. The joint

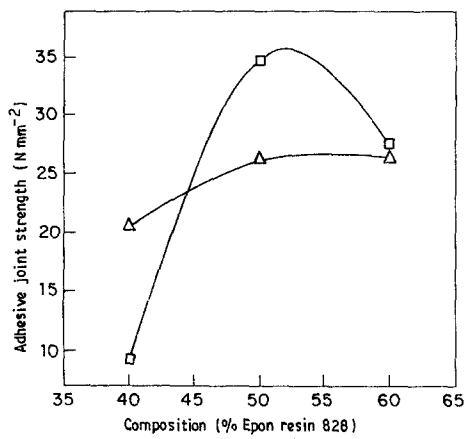


Figure 3 The effect of adhesive composition on the shear strength of joints subjected (□) and not subjected (△) before test to superimposed stresses at 1 Hz and 0.8 kg creep load at room temperature.

made with the EV-3 composition remained unaffected by this particular set of loading conditions.

It seems that the behaviour described above may be related to the glass transition temperature of the adhesive and the temperature at which the superimposed stresses were applied. As seen in Fig. 4, application of superimposed dynamic and static stresses at temperatures lower than the T_g of the adhesive led, for all the investigated compositions, to an increase of the joint shear strength. The same stressing experi-

ments performed at temperatures close to, or higher than the glass transition temperature of the adhesive (Fig. 5) cause a decrease in the joint shear strength.

The results presented in Figs 4 and 5 are plotted as normalized average shear strength values relative to reference specimens not subjected to stress prior to their testing in tension. Because of the application of superimposed dynamic and static stresses, a strengthening of the joints occurred over the whole range of torsional frequencies (0–15 Hz) applied to specimens subjected to both levels of creep load (0.8 and 6.4 kg). For the resin EV-2 (Fig. 4a) the strengthening was more pronounced when lower frequencies were applied and also at the same frequency when a higher creep load was used. The certainty of these tendencies of experimental data was tested by the Student *t*-test [44, 45]. This approach was necessary in view of the relatively high scatter of shear-strength data.

The lines in Fig. 4 were drawn to illustrate the general trend of the process. There was no intention to find, and they do not indicate, any mathematical relationship between the variables involved.

Fig. 5 shows the effect of similar superimposed dynamic and static stresses applied at higher temperatures (close to their glass transition temperature) on joints made with EV-2 and E compositions. It can be seen that, contrary to results observed at lower temperatures, the action of superimposed stresses in higher temperatures induced a general decrease of the

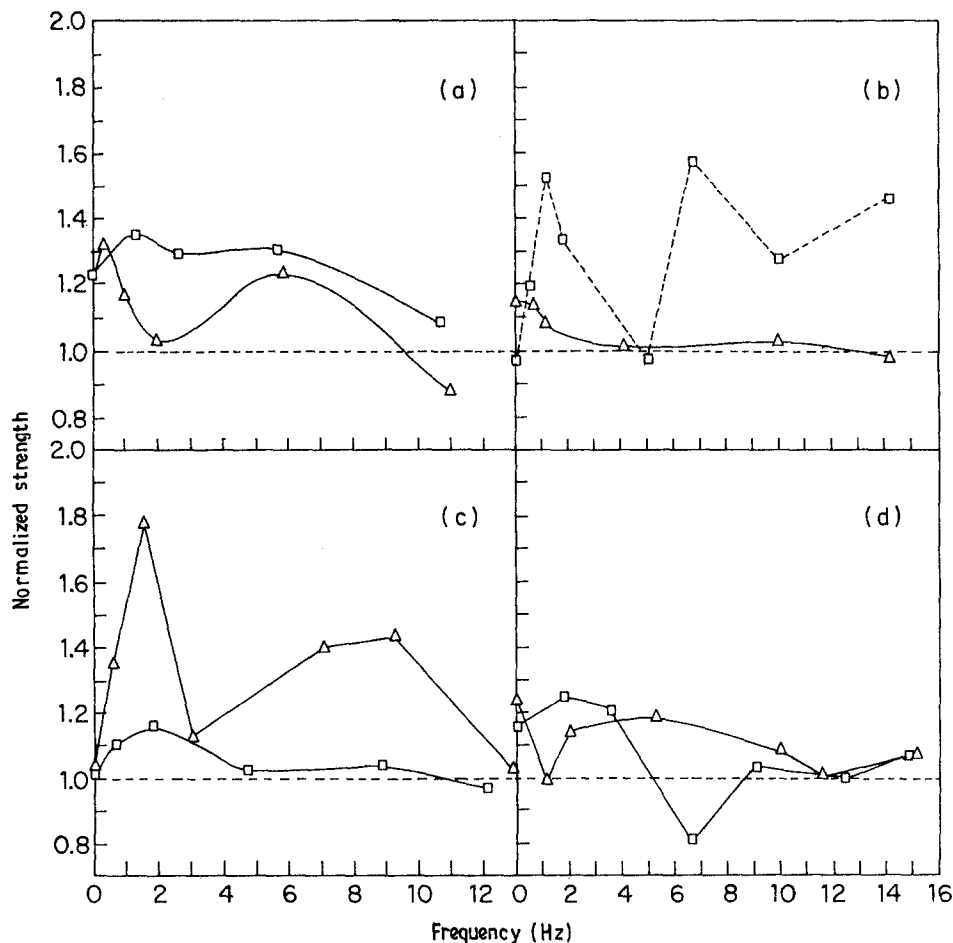


Figure 4 Normalized average shear strength of joints subjected prior to test to superimposed stresses: (a) specimens made with adhesive EV-2; (b), (c) specimens made with adhesive E; (d) specimens made with adhesive ET. The applied creep loads were: 0.8 kg per joint (△); 6.4 kg per joint (□). The superimposed stresses were applied at the following temperatures: (a), (b), (d) room temperature; (c) 64 °C.

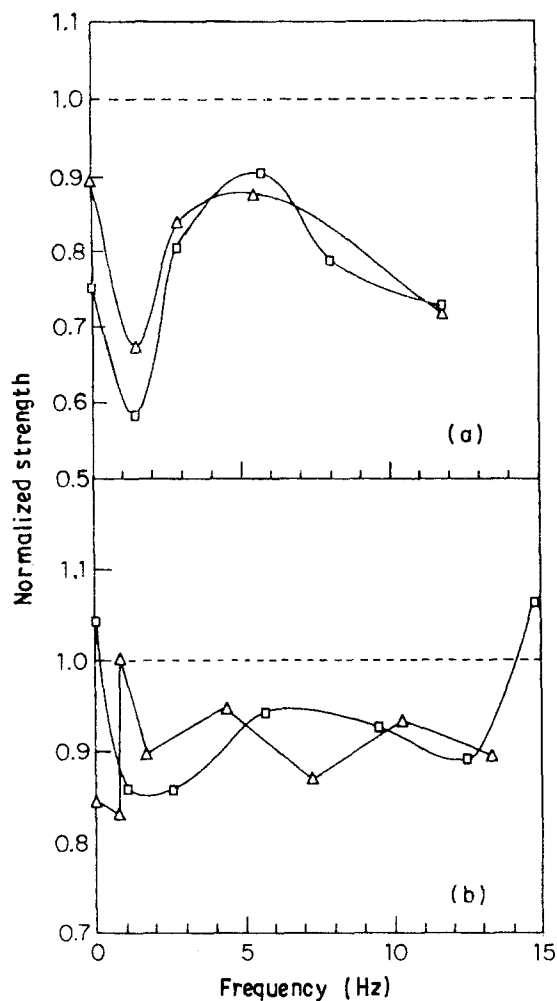


Figure 5 Normalized average shear strength of joints made with adhesives (a) EV-2, (b) E and subjected prior to test to superimposed dynamic and static stresses. The applied creep loads are as specified in Fig. 4. The superimposed stresses were applied at: (a) 52 °C and (b) 105 °C.

average shear strength. The decrease was more pronounced for the EV-2 composition: up to 40%.

Returning to Fig. 3 it is worthwhile mentioning that the EV-1 composition has a T_g of about 5.8 °C, which is lower than room temperature at which the superimposed stresses were applied; the shear strength of the joint measured at room temperature was indeed lower. The EV-2 composition has a T_g of about 52 °C, higher than room temperature at which the superimposed stresses were applied and consequently the shear strength of the joints increased.

Other interesting observations are connected with what seems to be a synergistic effect due to the applied static and dynamic stresses. It is known that the application of creep load alone may lead to toughening of epoxy resins and to an increase of the stress required to initiate crack propagation (static fatigue) [46]. Such an effect of strengthening due to the application of creep loads alone was indeed observed; on the ordinates in Fig. 4 are marked points representing the shear strength of joints subjected only to creep prior to testing. It was expected, based on the cyclic softening approach, that the superposition of cyclic loading will diminish this strengthening effect of the static load, but this was not the case. For a part (Fig. 4a and d) or for the whole range of torsional

frequencies used in our experiments (Fig. 4b, 4c, 5a, 5b), the superposition of cyclic stresses led to an additional increase of the joint's shear strength when compared with that of joints subjected only to creep. The effect was more noticeable (up to 20%) for the compositions with internal plastification (EV-2 and ET). The effect of superimposed stresses is more pronounced in the lower range of frequencies used in the study. This observation seems to agree with the findings of Marceau *et al.* [5] regarding the larger effectiveness of low-frequency vibrations in the reduction of the durability of adhesive joints made with epoxy resins.

Other authors recorded, during the first stages of fatigue experiments, an increase in the storage modulus (Schrager [47]) and of the density of the specimen (Rabinowitz and Beardmore [48]). These findings are similar in nature to the shear strength increase observed by us in joints subjected before the tensile test to a combination of dynamic and static superimposed stresses. The authors mentioned above also suggested that the heat generated during cyclic loading and the mechanical energy transferred to the specimen, were high enough to cause a continuous increase of "cross-linkages" in the sample. In their approach, these cross-links could be chemical (covalent) or physical (an increase of the amount of secondary bond interactions). We believe that the mechanism which governs the strengthening of joints following application of superimposed dynamic and static stresses is based on "physical" cross-links due to short-range orientation of network chains. A similar hypothesis regarding the intensification of intermolecular secondary forces due to reorientation was made earlier by one of the authors [49–51] when ageing of epoxy resins subjected to large strains was investigated. The possibility that this change is due to an increase of "chemical" cross-link density by continuation of cross-linking should be considered, but there are arguments which do not support this approach, at least in some cases.

First, no exothermal peaks were recorded in DSC tests performed after curing and/or application of superimposed stresses [53]; such peaks, if present, would indicate incompleteness of the cross-linking reaction [39, 40]. Second, the torsional strains used in our experiments were relatively small, thus resulting in the transmission of small amounts of energy due to cyclic torsion and low hysteretic heating, a fact which does not enhance further chemical cross-linking. Third, a decrease of T_g was registered in the second DSC run, when compared with the first run performed on the same specimen (adhesive E) in joints subjected to superimposed stresses at 105 °C [53]. Usually, as a result of cross-linking continuation during the first DSC measurement [52], the glass transition temperature would increase and consequently a higher T_g should be recorded in the second run. This was indeed the case with joints made with the same adhesive but subjected to superimposed dynamic and static stresses at a lower temperature [53].

As suggested earlier, more mobility seems to be provided to the network chains by means of the mechanical energy supplied by cyclic loading and this

presumably enhances short-range orientation of network chains under the acting creep load leading to an increase in the T_g of the polymers.

3.2. Morphology of the fracture surface

The effect of torsional oscillations, with frequencies of 0.38–11 Hz superimposed for 24 hours at room temperature on creep loaded (0.8 kg) joint specimens, on the glass transition temperature of the adhesive EV-2 is shown in Fig. 6. Micrographs of fracture surfaces obtained in tensile shear tests are displayed in Fig. 7. In spite of the fact that most of the investigated resins were at the testing temperature in their glassy state, the appearance of the fracture surface changes markedly from specimen I to specimen IV. The fracture surface of specimen I (Fig. 7a to c) is characteristic of polymers which failed in a brittle manner, namely fine radial river markings combined with a mirror-like appearance and a rough zone with very fine fibrils can be seen. These fibre-like structures detached from the surface seem to be formed through a brittle process of cleavage of the "fibre" from the substrate accompanied by plastic flow (Fig. 7c). The morphology of the fracture surface of specimen II (Fig. 7d to f) is similar to that of specimen I, but a larger smooth surface can be seen and the structures on the zone with radial markings seem more dainty (compare Fig. 7b with Fig. 7e). The "ladder" appearance is generated during the stick/slip crack propagation, a result of plastic processes occurring at the crack tip (Fig. 7e, f and i). When joints subjected before test to a torsional frequency of 2 Hz (specimen III) are fractured in the tensile experiments performed at room temperature, they exhibit a completely different fracture surface than the previous ones. The failure surface loses its specific brittle appearance and becomes less featured, large curved structures appear instead of the "ladder" ones (Fig. 7g and h), and the arrest lines are wider and warped with triangular markings. A very delicate structure, occurring at an angle of about 90° , can be

observed on the primary fracture structure and signs of plastic flow are visible at the base of the ridges (Fig. 7i). In specimen IV the fracture surface is less featured and the smooth area is much larger. The plastic deformation is visible now even at lower magnification in the shape of flow-like features close to the substrate (Fig. 7k) and at higher magnification the tip of the crack appears open with very clear plastic features.

The above description of failure surfaces of the adhesive joints fractured in shear agrees with observations made by other authors who studied the fracture of similar resins in their bulk form at increasing test temperatures within the glassy region. Morgan *et al.* [27] investigated the tensile fracture behaviour of different amine and polyamide (Versamid 140) cured epoxides. They found that a temperature increase in the sub- T_g range leads to extension of the areas of smooth, mirror-like fracture topography. At temperatures near T_g these smooth zones spread over the whole fracture surface. More recently, using image-processing techniques in combination with optical and scanning electron microscopy, Cantwell *et al.* [14] confirmed the above findings. They examined anhydride-cured epoxy resins with various specimen geometries and found that the radius of the smooth zone increased six-fold when the temperature increased in the sub- T_g range by 80°C .

In the present work, a similar result was obtained by maintaining the test temperature constant, while the glass transition temperature of the adhesive was changed by alteration of experimental parameters (Fig. 6). The increase in the smooth area of the fracture surface observed in the present work is like that reported by Morgan *et al.* [27] and Cantwell *et al.* [14]. The values of T_g of the adhesive and the joint strength decrease from specimen I (Fig. 7a to c) to specimen IV (Fig. 7j to l). The brittle aspect of specimen I can be explained as follows: the adhesive EV-2 with its higher T_g is, at room temperature, at a lower correspondent temperature ($T_{\text{corresp}} = T_{\text{exp}} - T_g$) than other specimens tested at the same temperature which have lower glass transition temperatures. Based on this reasoning, the gradual changes in the fracture morphology (i.e., the vanishing of the brittle fracture features and the increase of plastic flow phenomena including the growth of smooth surface areas within the fracture surface) can be expected. The plastic flow marks visible in our micrographs were reported also by others in their fractography studies of bulk polymer specimens and even confirmed by birefringence studies [26, 27].

When the fracture behaviour of single lap joints is investigated, the picture is more confused than in the case of bulk fracture because of the very complex state of stresses acting within this type of joint in tensile loading [54, 55]: normal and tensile stresses are combined with shear stresses in different ratios across the overlap area. In addition, the small thickness of the adhesive layer can emphasize the effect which the organic and inorganic parts of the interphase may exert on the fracture behaviour of the small bulk of the resin in the joint. The crack initiation takes place

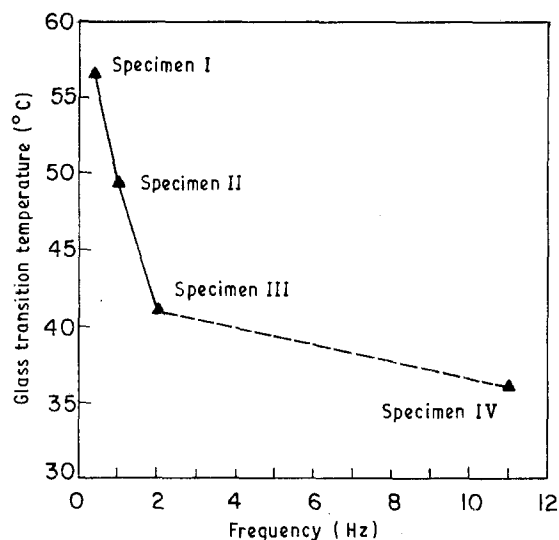


Figure 6 Frequency effect on the glass transition temperature of EV-2 adhesive. The joints were subjected before test to creep load of 0.8 kg per joint at room temperature combined with torsional oscillations at frequencies shown in figure.

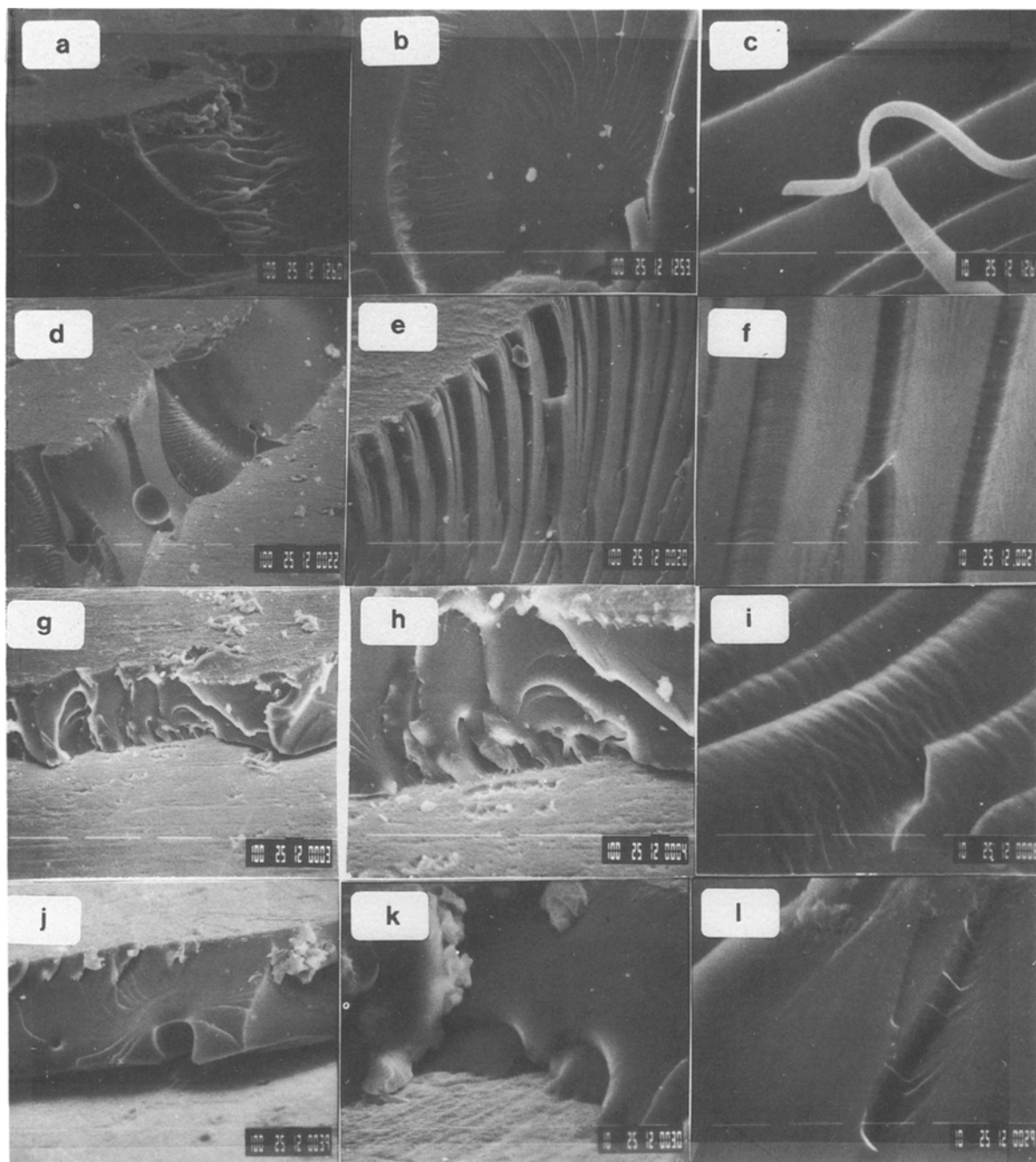


Figure 7 SEM micrographs of fracture surfaces of joints subjected prior to tensile shear test, to creep load of 0.8 kg per joint at room temperature combined with torsional oscillations at frequencies: specimen I – a, b, c – 0.38 Hz; specimen II – d, e, f – 1 Hz; specimen III – g, h, i – 2 Hz; specimen IV – j, k, l – 11 Hz. The T_g of various specimens are shown in Fig. 6. The bars in SEM micrographs have the length (in microns) equal to the first number in the black rectangle (at the bottom right of each picture).

simultaneously at different locations in the interphase defects and air bubbles, and the fractography is expected to be very complex. Several cracks will propagate simultaneously in different directions thus resulting in a failure surface not organized as well as that in bulk specimens produced especially for fracture studies.

3.3. Fatigue

A feature common to the adhesive layer and to the bulk form of the same resin can be found in the fatigue failure of the polymer. When the amplitude of torsional oscillations was larger at a temperature close to the glass transition of the adhesive, all the joints failed

during the superimposed stressing experiments, usually within the first two hours. Even at low amplitude, a few joints failed during the experiments in cases in which the temperature was close to T_g . In both situations, the fracture surface has a characteristic aspect seen also in the fatigue failure of bulk thermoplastic and thermosetting specimens, namely many parallel, fine stria and furrows can be distinguished [56–62]. These phenomena present on the failure surface of EV-2 joints, are shown at different magnifications in Fig. 8. The distances between the striations and furrows ($0.5\text{--}2\ \mu\text{m}$) do not remain constant, but increase in the direction of crack propagation. Their occurrence is presumably due to the intermittent progress of the crack each time for one or more cycles [63]. The

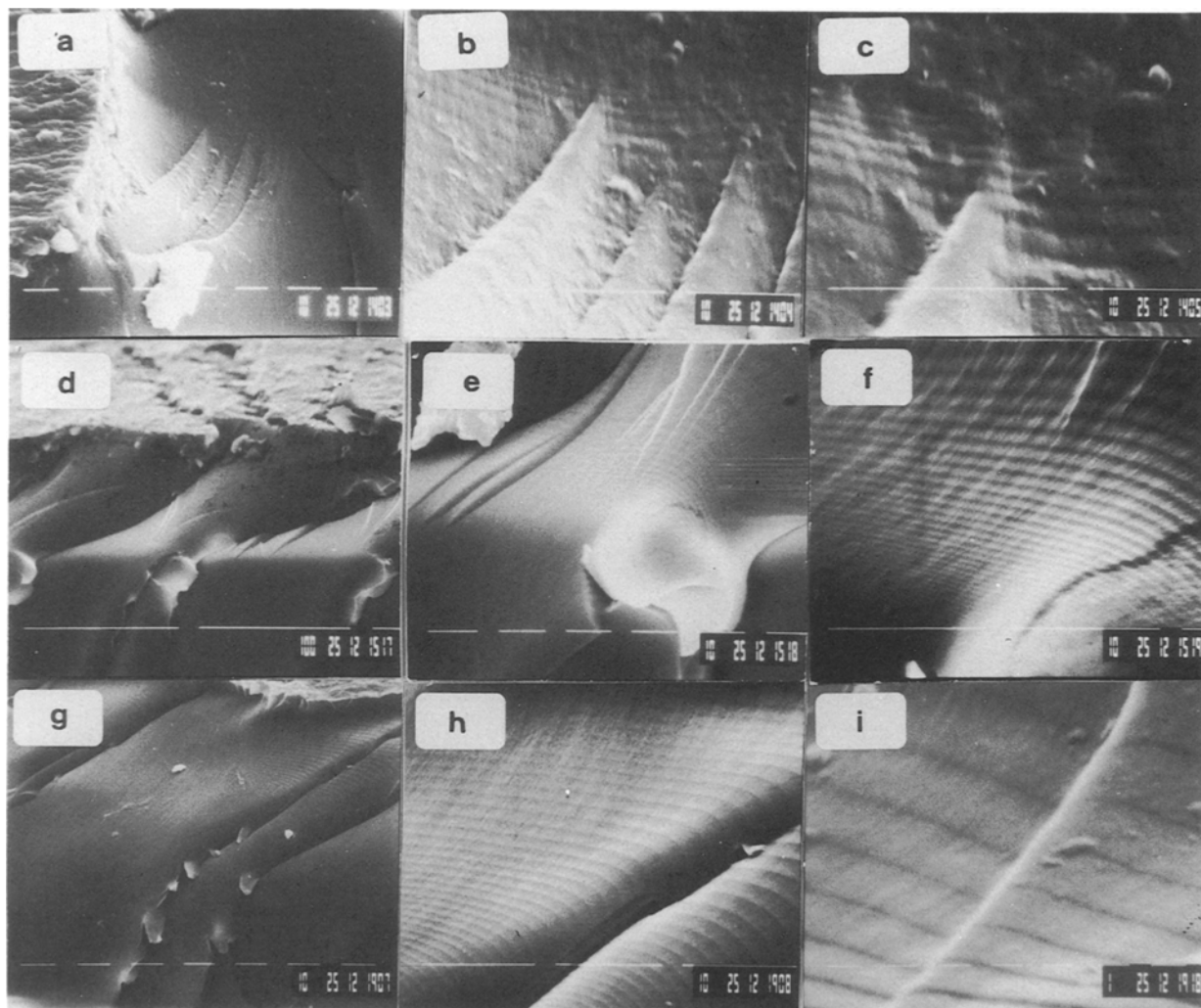


Figure 8 SEM micrographs of fracture surface of joints made with adhesive EV-2 after fatigue failure during application of superimposed dynamic and static stresses at 52°C. The experimental conditions: a, b, c – frequency 11.8 Hz, creep load 0.8 kg; d, e, f – frequency 11.8 Hz, creep load 6.4 kg; g, h, i – frequency 1.45 Hz, creep load – 6.4 kg, torsional amplitude about three times larger than in previous specimens; the bars length – see Fig. 7.

mechanism for this periodic advancement can be explained by the plastic deformation taking place at the crack tip [62, 63].

4. Conclusions

1. A simple combination of dynamic and static stresses, as used in the present work, affects considerably the mechanical behaviour of a model joint. The implementation of new methods, which will involve combinations of such stresses in the process of characterization and testing of adhesive joints, appears to be mandatory in reducing the existing gap between laboratory testing results and joint behaviour in conditions closer to the real ones.

2. Significant increase of the shear strength of joints prepared with three different epoxy resins occurred when, prior to their strength testing, the joints were subjected to simultaneous action of torsional oscillations and creep shear at temperatures below the T_g of the adhesive.

3. Combinations of stresses applied at temperatures close to, or higher than, T_g resulted in a reduction of the shear strength of the specimen, as

compared with the shear strength of reference specimens.

4. Experimental evidence shows that the superposition of torsional oscillations on specimens subjected at the same time to shear creep stressing below the T_g of the adhesive, led to an additional increase of the joint strength. Based on the above observation and on some existing literature data, it can be assumed that strengthening of the joint as a result of superimposed dynamic and static loading may be due mainly to short-range orientation of network chains and to an increase in intermolecular interactions between highly polar sites of the investigated network [49–51]. The possibility of changes in chemical cross-linking should not be discarded.

5. A qualitative correlation can be found between changes occurring in the properties of the adhesive as described earlier and the morphology of the surface created during the joint fracture. It is suggested that changes in the fracture morphology of the joints are related to the variations occurring in the T_g of the adhesive. These changes are visible even if the failure of the joints takes place in sub- T_g temperatures of the resin. The fracture surface shows a significant increase

of plastic features with the lowering of the T_g of the adhesive.

6. At large amplitudes of torsional oscillations and/or at temperatures close to the T_g of the adhesive, fatigue failure took place and a specific, striated and furrowed morphology was recorded. This fracture behaviour is common also in the bulk form of resins which failed by fatigue. Because the crack propagates in an uncontrolled manner within the single lap joint and it is hard to orient the fracture surface with relation to the electron beam, it was impossible to find any correlation between the thermo-mechanical history of the joints and the dimensions of the fatigue furrows as seen in SEM micrographs.

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