Resource A

PAPER 3

Premature fracture of a composite nylon radiator
Premature fracture of a composite nylon radiator

P.R. Lewis

Department of Materials Engineering, Faculty of Technology, The Open University, Walton Hall
Wilton Keynes MK7 6AA, UK

Received 30 August 1996; accepted 8 September 1996

Abstract

Fracture of a GF nylon composite radiator occurred in a new car, leading to seizure of the engine. The fracture probably started at a cold slug or void present on an unusually large weld line in the radiator itself, probably created by poor moulding conditions. Rather than being a design fault, the failure was probably caused by lack of quality control during injection moulding. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Radiator; Nylon; Composite; Void; Weld line; Fracture

1. Introduction

A new design of radiator tank failed on a new car during test driving. The tank was constructed from glass-filled (GF) nylon, a composite material used in engine compartments for its temperature resistance and strength. Many inlet manifolds, such as that on the new Jaguar XK8 for example, are now made from GF nylon 6,6 using the hot-injection injection moulding process [1].

The car had only travelled about 500 miles before catastrophic failure of the cooling system, which led to seizure of the engine. Some 200 similar prototype tanks had been produced and fitted to similar cars, and the manufacturer was concerned that there might be a design problem. Although they had considerable experience with the material in other radiators, the bodies were moulded by a sub-contractor elsewhere.

They therefore wished to know how the crack had been formed in the radiator, and whether the problem was due to faulty material, poor design or manufacture, or a combination of such causes. A programme of microscopy was undertaken to examine the fracture surface and other features of the moulded tank. A new, unused tank was used for comparison. Mechanical testing was also used to examine the quality of the material.

* Tel. 01908 635378, Fax. 01908 63530

© 1999 Elsevier Science Ltd. All rights reserved.

ISBN 0444 5311 - see front matter - 1999 Elsevier Science Ltd. All rights reserved.
2. Survey of failed whole radiator

The failed part was examined for its surface quality first, and key features then examined with an optical microscope. SEM was used to resolve details of interest.

2.1. Macroscopic inspection

The radiator comprised a single moulding (Figs 1 and 2) with a centre gate, judging by the large sprue remnant in the centre of the underside (Fig. 3). The clean appearance of the sprue suggested operator cut-off, its relatively large diameter of ca 1 cm being necessary to allow the high viscosity

Fig. 1. Failed and new radiator boxes compared. The upper, failed sample cracked after 500 miles in service.

Fig. 2. Comparison of lower ends of upper, failed box and new box below. Closed arrow shows brittle crack which ran along inner corner of adjoining fan buttress. Open arrow shows contamination from leaking cooling water when tank was in situ.
glass-reinforced nylon 6.6 compound to enter the tool cavity smoothly. The failed tank is compared directly with a new moulding taken from the same batch in the three figures. A small amount of carbon black had been added to the compound to give a matte black colouration. Both tanks were date stamped, indicating that they had been moulded only recently.

Direct comparison of the tanks showed the failed tank to be distorted along its greatest axis, the sidewalls bulging inwards, as shown in Fig. 3. Such distortion can be caused by relaxation of internal frozen-in strain developed during moulding at temperatures below normal, or low melt temperatures in the barrel of the moulding machine. The tank had experienced only a few cycles from ambient temperatures and pressures up to working conditions in excess of 100°C and 25 psi over atmospheric pressure. Such conditions allow internal chain orientation to relax to the equilibrium state owing to the extra thermal energy provided for diffusion.

2.2. The crack and adjacent features

The single crack which had led to loss of water pressure and loss of cooling action for the engine, was situated near an external buttress, used to support a nearby fan. Tidemarks were visible immediately next to one end of the crack, their position showing the tank to be placed in a vertical,
Fig. 4. Macrograph of brittle crack, running along stress concentration of buttress corner, and ending at points shown by closed arrows.

Fig. 5. Macrograph of brittle crack on inner surface, with ends shown by closed arrows. Weld line at left (open arrow) co-linear with crack, and surrounded by extensive flow line pattern.

upright position when in use on the car. The crack was brittle in nature, and extending ca 6.3 cm internally and almost the same distance externally (Figs 4 and 5).

The external surface was clear of any other major defects, and no defects were at first apparent on the inner surface owing to a superficial deposit from the cooling water system. On gentle rubbing, however, very clear traces of flow line patterns could be seen over much of the inner surface. Such patterns were revealed because the ends of the glass fibre reinforcement tend to roughen the otherwise smooth surface, and they also tend to be aligned with the melt flow, so will collect particles and show any major changes in fibre or melt orientation. Figure 5 in particular,
shows a serious weld line surrounded by an extensive flow line pattern, the weld line leading directly into the crack.

The flow pattern could also be traced further away from the crack (Fig. 6). It appeared to emanate from the sprue, and was aligned towards one of the far corners of the box. A defect found close to the sprue, comprised a deep, short irregular weld line often known as a 'cold slug'. Such defects are generally caused by incomplete melting of the moulding pellets, whose external shape is thus partly preserved in the melt (Fig. 6).

Whiting gently rubbed into the inner surface of the new box revealed a flow line at a very similar position, under the fan buttress. However, not only was this flow line less severe, it was also clear that the overall flow pattern thus shown was quite different to that in the failed box. In particular, there were no cold slugs, and the flow pattern was absent near the sprue.

2.3. Etching experiment with new tank

New tanks of slightly different design, but made from the same material, were used to measure the intrinsic strength of the material as well as investigate the internal structure of the moulding. A new tank was sectioned and polished for microscopy. The exposed section was etched with chromic acid, a method which reveals internal structure by selectively removing the polymer matrix (Figs 7 and 8). Etching revealed first, voids ranging in size from ca 0.3 mm (or 300 μm) to less than 20 μm in diameter. The largest voids were detected in the centre of the thick edge section (Fig. 7), the smallest visible at this scale tending to occur more widely in the centre of the thinner wall section (Fig. 8). The etchant also revealed changes in fibre orientation, especially evident in the region between thin and thick sections of the edge (Fig. 7), but also present elsewhere in both specimens. The effect is caused by changes in orientation of the polymer melt, since the glass fibres tend to align themselves with the laminations of the melt as injection into the tool cavity occurs during hot moulding. The short fibres tend to align parallel to the surfaces of the tool, where
parallel laminar flow occurs, but tend to tumble towards the interior, where the polymer laminae are folded into a much more complex pattern. Here, the orientation of the fibres is much more random, so the light reflected from the section is lighter in tone. On the other hand, light is absorbed by preferential orientation of the fibres at the surfaces. The effect is generally known as the 'skin/core effect' [2].

Unfortunately, the ideal tends to break down when real moulding sections are examined in detail. The skin/core effect was seen at its best, ideal form at the left-hand part of the thinner section (Fig. 8). Here, there was a clear skin approximately 0.5 mm thick on both inside and outer
Fig. 9. X-radiograph of side of new tank (top) and part of failed tank (bottom). Trace of flow lines can be seen in the upper radiograph (between the open arrows). The crack is well shown in the lower section (solid arrows). The thick lower edges of both radiographs show variable density along their length due to internal voids.

surfaces, but it increased in size towards the middle part of the section, and finally broke up into a more complex region on the right-hand side of the figure. The oriented skin appeared to be much thicker in the edge section of Fig. 8, and the skin/core effect less clear cut towards the right-hand part of the thinner wall abutting the edge buttress. The voids tended to be more prevalent in the randomly oriented core parts of the sections, especially in thicker parts of the moulding.

2.4. Radiography

Some of the sections were radiographed using soft X-rays provided by a medical source [3, 4]. They showed the critical crack in excellent detail, and also provided evidence of the flow lines and clumping of fibres seen in the etched sections (Fig. 9). One shot from the failed tank, showed the faint trace of a 'cold slug' near the sprue. It reinforced an earlier observation (Fig. 6), giving an important clue to the cause of failure, because it indicates incomplete melting of the granules used to feed the injection moulding machine.

3. Microscopic examination

It was important to examine the fracture surface, for determining the crack morphology. Since the crack was trapped in the solid side of the tank, it was necessary to break the material in a
controlled fashion so as to liberate the crack surface. The process was tackled in two stages, first involving cutting along the main corners in the failed tank, so as to produce a ‘lay-flat’ set of samples. One interesting result of this procedure was that the outward bulging in the whole tank was reversed, so that the sides bulged inwards (cf. Fig. 3). It was also noticed that the material everywhere in both samples proved rather brittle, as perhaps what one might expect from the high filler content of 30% glass fibre.

3.1. The fracture surface

The second part of the procedure involved liberation of the crack (Fig. 10). The fracture surface was later plated with gold for SEM. This allowed detailed inspection of the 10 \( \mu \text{m} \) diameter fibres present (Fig. 11). The gold treatment was also helpful in enhancing the contrast for optical microscopy (Fig. 12).

3.2. Tidemarks from the leak

The side of the external buttress just by the critical crack showed several stains produced by escape of cooling fluid, and comprised a brown tide line underlying a set of white tidemarks (Fig. 10). The white marks indicate a series of small contamination incidents, possibly five or more before final failure. Each may mark a point when the crack or cracks connected the inner reservoir
Fig. 11. Various SEM shots of fracture surface.
Fig. 12. Panoramic sequence of gold-plated fracture surface, left to right across crack from top to bottom. Remnants of cold slugs and lower weld line shown by open arrows.
to the exterior, so allowing seepage of cooling fluid to the outer surface of the tank. This occurred
during use of the car, so pressurising the cooling system, subjecting the outer skin to tension and
initiating cracks. It suggests that the several cracks propagated until penetration to the reservoir
occurred, leading to small spots of fluid being ejected onto the adjacent buttress.

It is tempting to suggest, judging by the size of the brown stain, that the initial leak occurred
just by the left-hand corner of the buttress (Fig. 11). No necessarily so. The tank in use is vertical,
so if leaks were occurring anywhere above this corner, leak would tend to collect here as a band
since it would be adhering to the corner created by the buttress and the adjacent tank surface.

So what defects were visible on the fracture surface? One feature was the several smooth,
irregular zones; most clearly seen in the optical micrographs of Fig. 12. There were several areas
where such features occurred: a smaller cluster under the first buttress corner, a group near the
second buttress corner and a linear, shallow zone on the underside of the fracture surface. The
irregular form of the first two groups suggested that they may represent fragments of the original
pellets used in the moulding process which have not fused together, and thus represent lines of
weakness within the solid material. They could thus be most closely related to the cold slug defect
found on the inner surface of the moulding, near to the sprue (Fig. 6) and one of the radiographs.
The linear zone was the clearest indication of a 'true' weld line, which would be formed when the
pellets have lost their original shape due to melting, but then two streams of molten plastic have
impinged without fusing. The smooth areas in the interior could also represent internal weld zones.

4.3. SEM examination of fracture surface

High magnification SEM examination of an area near the first buttress showed a widely varying
microstructure [Fig. 11(a)]. Some areas appeared free of fibres, while others possessed a dense
distribution of broken fibre ends. Fibre orientation in the area below the left-hand corner of the
first buttress appeared to be uniform, and oriented to the buttress and neighbouring external
surface. At a slightly higher magnification, Fig. 11(b) shows the virtually fibre-free part at the inner
edge of the fracture immediately below the first buttress corner. A crack branching directly into
the bulk delineates the internal edge of this feature, which represents the linear weld line mentioned
above. The smooth surface of this zone contrasts sharply with the very rough surface immediately
above, where numerous fibre ends protrude from the surface. Voids may be present just above this
zone. The final plate [Fig. 11(c)] shows the lower weld line next to the inner surface below the
buttress.

So where did the cracks start? There are numerous points or zones which could represent origins;
the most likely positions are the two zones near to the corners of the first buttress, which is a fairly
severe stress concentration, where extra stress magnification will have been created by latent defects
such as voids, cold slugs or weld lines.

4. Mechanical tests on tank material

It was important to conduct some simple tensile tests on bars cut directly from both new tanks
and the failed tank to determine the intrinsic strength of the material, in both the failed and new
The ideal samples to examine would be those cut across suspected weld lines, since then the defects would be subjected to the highest stress when the samples were pulled in tension.

Four samples were cut, two from the new tank in a region showing serious flow lines, and two from the failed tank showing similar flow lines, but well away from the failure crack. One of each pair was cut parallel to the flow lines, the other at right angles to the flow lines. The samples were about 5 cm long, and possessed a ca. 1 cm narrow central region about 5 cm long, so giving a dumbbell shape. The samples were cut very carefully using a small hacksaw along the pre-marked shape, and polished by hand with a series of finer emery papers to remove any edge imperfections which could cause premature fracture.

All the specimens were tested at room temperature (ca. 25°C) and ambient humidity (ca. 50%), using a constant strain rate of 0.1 mm s⁻¹. Three of the four samples broke centrally, the fourth (No. 2) on the edge of the shoulder. All the samples broke in a brittle fashion, i.e. the two parts of the fracture surfaces could be fitted back together. The results were as follows:

- Sample No. 1 (new, lateral) \( \sigma_b = 84 \text{ MN m}^{-2} \)
- Sample No. 1 (new, parallel) \( \sigma_b = 81 \text{ MN m}^{-2} \)
- Sample No. 3 (old, parallel) \( \sigma_b = 80 \text{ MN m}^{-2} \)
- Sample No. 4 (old, lateral) \( \sigma_b = 55 \text{ MN m}^{-2} \)

The elongation to break was very similar for the first three samples, at approximately 9%, and 10% for the final sample.

It may be noted that the best result of 84 MN m⁻² calculated for sample No. 1 fell somewhat below the ideal value given in the data sheet for this polymer (140 MN m⁻²). Sample No. 3 appeared to have failed from a surface tool impression mark, present as a sharp corner across part of the dumbbell, for example. Inspection of the fracture surfaces, however, showed them to be reasonably free of internal voids and cold slugs of the kind found in the fracture surface (Fig. 12). All the fracture surfaces showed a central 'spine' or cusp indicative of skin-core control of fracture, quite unlike that of the critical fracture. The calculated elongations to break are rather greater than the value of 6% quoted on the same sheet, but strength and elongation to break being given for samples conditioned to ambient temperature and humidity essentially identical to those used here. Comparison of the tensile strengths showed that the material from the new tank is superior to that from the failed tank. Although there were visible flow lines in all the samples, failure did not seem to be related to them in any clear, unambiguous way. In general, the tests reinforced earlier impressions when cutting the tanks for analysis of relatively stiff but brittle mechanical behaviour. The addition of 30% chopped glass fibre improves the stiffness of nylon, but at the expense of strength.

5. Discussion

A reasonably clear picture of the failure emerged as a result of detailed examination of the critical fracture surface, and comparison of the properties of new and unused tanks. The critical crack probably resulted from the coalescence of several smaller cracks below the fan buttress.
There were several types of defect seen in the fracture surface here, including a large weld line, cold slug fragments and voids. The latter are especially likely to form at or near a thickening of the tank wall (e.g. Fig. 7). Judging by the several white stains observed on the buttress surface immediately above the lateral corner, it is likely that there were several very slow leaks before the final, catastrophic failure when the cracks propagated catastrophically. The head would have formed during this phase of the failure. It was possible to quantify the effect of the several weakening mechanisms at work in the failure. The initiating mechanisms are:

1. the geometric stress concentration at the buttress corner;
2. internal voids;
3. fragments of cold slug adjacent to the corner; and
4. frozen-in strain due to cold tool or cool melt conditions when moulded.

The maximum tensile stress to which the outside of the side wall is exposed can be estimated by simply assuming that the tank can be modelled by a cylindrical pressure vessel. The maximum stress developed in such a vessel is the hoop stress, which acts around the short periphery of the tank, at right angles to the long axis of the tank. It is the most serious stress experienced by a cylindrical pressure vessel or tube, and is twice the longitudinal stress. It is reasonable to use the hoop stress as the critical stress imposed on the tank, since the crack has propagated in a longitudinal direction, i.e. under the influence of a hoop stress. It is given by the equation

\[ \text{Hoop stress} = \frac{pD}{2t} \]  

where \( p \) is the internal pressure, \( D \) the diameter of the cylinder and \( t \) the wall thickness. Taking \( D \) as ca. 45 mm and \( t \) as 2.5 mm together with the value of \( p \) as 25 psi, then the hoop stress is thus about 225 psi or about 1.55 MN m\(^{-2}\), a relatively benign stress for a material with a measured tensile strength of ca 80 MN m\(^{-2}\).

The stress concentration at the buttress corner can be modelled by a standard figure provided by Peterson [5], which represents a notch in bending for various geometries. Inserting the measured values for the three geometric parameters. \( r \), the radius of curvature at the corner = ca 0.01 mm; \( d \), the thickness of the section = 2.5 mm and \( D \), the thickness of the buttress = 30 mm — then the critical ratio for interpolation on the graph are \( r/d = 0.004 \) and \( D/d = 12 \).

Using the value of \( r/d \) of ca. 0.004, then interpolation gives the stress concentration factor, \( K \), (the ratio of real to nominal applied stress) as

\[ K = 4.2 \]  

If the spherical void occurs in this zone, then the \( K \) value will be about 2, but it is likely to be an underestimate, since they vary greatly both in shape and inter surface. If flatter and elongated, then a more realistic model is that of a penny-shaped crack [6]. The \( K \) factor varies from about 2 up to greater than 11 on this stress concentration diagram, depending on the ratio \( r/t \), where \( t \) is the minimum radius and \( r \) the radius of the circular cavity. This was the most difficult parameter to estimate since direct measurement was difficult and impracticable with the available microscopic data. Taking a pessimistic value of say, \( r/t = 20 \), then the \( K \) value will be about 6, so the net stress factor could be

\[ K = (6 \times 4.2) \approx 25 \]
The third factor, the frozen-in strain, produced a visible widening of the original crack of ca 0.5 mm over a diameter of approximately 50 mm. This is equivalent to a strain of approximately 1%, or approximately 0.5 kN load, equivalent to a stress of approximately 20 MN m\(^{-2}\) by interpolation on the load extension curve. So the total resultant (pessimistic) stress is given very approximately by the equation

\[ \text{Total stress} \approx (25 \times 1.55) + 20 = 50 \text{ MN m}^{-2} \] (4)

This value may be compared with the best experimental estimate of about 80 MN m\(^{-2}\), and with a lower bound of about 55 MN m\(^{-2}\) for the inherent strength of the composite nylon material.

The argument may thus be summarised. The combined effects of a geometric stress concentrator at the corner of the adjacent buttress and cracks (either present as a void or at the surface of a cold slug or weld line just below the corner), effectively magnified the real stress experienced by the material by some 25 times. The material of the tank was also in a state of strain produced by cold moulding, so that an extra component of about 20 MN m\(^{-2}\) must be added to the magnified stress, giving a total stress of about 59 MN m\(^{-2}\). This value is comparable with the mean strength measured for the material, and exceeds the lowest value actually obtained. It thus becomes possible to see why cracks were initiated near the buttress corner and grew intermittently with each successive pressurisation of the tank. Crack growth would, of course, have accelerated with each successive exposure, since the length of the crack and hence the magnification at the tip would have grown in step. The last event would probably have been the worst, and the event which directly caused catastrophic leakage of cooling fluid, before the crack re-stabilised, owing to relaxation of the frozen-in strain (Figs 3 and 6).

It is finally important to point out that no allowance has been made in this calculation for the extra stresses imposed on the tank through the buttresses and lugs by fitment of fans, motors, coolers etc. Similar considerations apply to the inlet and outlet pipes, especially as they will be stressed by fitment of connecting tubes. All such add-ons will of course exacerbate the situation.

The possibility that the rogue moulding was produced during the warm-up period of the injection moulding machine, remains the most likely cause of the failure.

6. Conclusions

1. A failed radiator tank has been examined in detail for the origin and causes of its rapid catastrophic fracture on a new car. The crack was brittle in nature and had started at or near a corner buttress. It propagated in several steps, probably corresponding to intermittent use of the car, and exposure of the radiator to a normal, expected hydrostatic internal pressure of 25 psi. This pressure is equivalent to a sidewall stress of only about 1.55 MN m\(^{-2}\), well within the ca 80 MN m\(^{-2}\) strength of the material.

2. This benign stress was magnified, however, by a combination of three factors, two of which are related to the moulding conditions under which the product was made, and the third is related to the design geometry of the tank. The first factor was the presence of cold slugs or unmelted or partly melted material in the outer sidewall, probably caused by moulding into a cold tool or using too cool a melt. A larger, similar slug was discovered near the sprue, but had not led to failure since it was not near to the second factor involved, a geometric stress concentrator.
The failure cracks started from cold slugs or voids, because they occurred near the base of an adjacent buttress, which possessed a sharp corner with the sidewall, and represented a stress raising factor of about 4.2. The cold slugs and/or voids were more serious in their stress magnifying effect, possibly having a stress raiser of about 6, so the effective stress concentration in this zone was about 25 times the nominal applied stress of only 1.55 MN m⁻².

3. The final factor which made the situation critical was the presence of a substantial level of frozen-in strain, produced by cold moulding the material. This effectively added some 20 MN m⁻² extra stress to the surface of the cold slug, producing a total stress in the region of about 59 MN m⁻², a stress comparable with the mean strength of the material. Brittle cracks were thus initiated at the surface of cold slugs or voids when the system was first pressurised, and grew progressively at each use of the cooling system. The cracks had penetrated through to the interior of the radiator, but only small quantities of water leaked out under pressure, judging by the several traces of contaminant found near the crack. When the cracks reached a critical size, they propagated catastrophically, releasing pressure from the system, and hence resulted in loss of the cooling facility.

4. The failed specimen appears to be a maverick which was probably accidentally included in the batch sent to the car manufacturer. Normal QC procedures usually prevent such mouldings being used, but when this batch of tanks was moulded, they failed to catch the rogue product. Careful visual inspection at the moulding machine would probably have caught the rogue, provided the operator was aware of what to look for in terms of defects such as weld lines and cold slugs. It is a difficult product to examine quickly for such defects owing to its black colouration, which were only revealed by dusting with whiting and by very close visual inspection.

Acknowledgements

Naomi Williams (OU) for SEM, Jim Moffatt (OU) for mechanical testing, Gordon Imlach (OU) for etching experiments, and the manufacturer for permission to publish this edited account of a more substantial report.

References


[6] Peterson, op cit., Figure 150. Pilkey, op cit., Chart 4.76.