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A Survey of the Development of Contour Etching with Particular Reference to the Effects on Tensile and Fatigue Strength of Alclad Alloys of Aluminium

- by -

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SUMMARY

Present methods of contour etching used by aircraft manufacturers in England and America are described and compared. Experiments to develop a production technique are described and factors controlling the process and product are discussed. There is no significant change in the tensile strength even when some 80 per cent of the original thickness is removed. It is doubtful if the etching process 'per se' reduces fatigue strength and the difficulties of making an analysis of effects on a clad material are discussed.

The considerable advantages of the process in the production of aircraft are shown in some detail.

CONTENTS

FIGURES

1. History, description and advantages of contour etching

When a metal is immersed in a suitable chemical reagent, usually an acid, it will be eroded fairly uniformly, but if an area is covered with a resistant coating, this locality will be protected whilst metal is removed from the unprotected areas. The method has long been used in the printing industry for the production of copper blocks. The modern process of Contour Etching (or Chemical Milling) uses this effect, with the necessary modifications and controls, for the production of structural shapes by other methods would be uneconomical or impossible. The process has been applied (Refs. 1 and 2) to alloys of aluminium, magnesium, titanium, brasses, carbon and stainless steels. There are few limitations to the intricacies of the surface pattern. Its depth may be controlled by the etching time and may be stepped by successive operations. The product of a tapered section of large area is very difficult by machining but with Contour Etching the component can simply be lowered into the etching reagent at a controlled rate. Forgings or extrusions may be reduced locally or overall, though some local attack may take place at outcropping layers of inclusions. Components may be stretch formed, rolled or pressed and then etched to final shape and dimension.

The process is a new technique for the shaping of large areas of metal and is particularly useful in the production of intricate shapes. It is claimed that the rate of removal of metal is competitive with machining in many cases.

2. Advantages of Contour Etching for the manufacture of aircraft

Design and weight saving

Large areas, such as wing surfaces, always present an expensive problem for machine tools, but such components are ideal for contour etching.

The design of large thin components with the minimum number of joints can be proposed. Unwanted metal, in the many places which would not be accessable to machine tools, can be removed and the possibilities of integral construction can be explored. A weight penalty need no longer be suffered where design thickness is less than standard sizes. Closer tolerances (0.002") can be called for on skin thicknesses (Ref. 3) than normal machining allows so more accurate stress analysis is possible. Better advantage can be taken of sandwich construction by recessing the honeycomb into the skin to improve the bond. The minimum web thickness available in a forging is not so restricted since local etching is possible where the part could not be machined.

Since there is less scatter on the S-N curves for contour etched material than on those for machined material, (para. 5.2.2.) the allowable stress on a structure subject to fluctuating loads is more accurately determinable and therefore a lower factor of safety may be employed.

A contour etched surface usually has a better finish than a machined surface, and is more uniform. There is less need of a polishing operation to remove machining marks.

The principal disadvantage of the process is that sharp internal corners cannot be produced. However, they are rarely called for and can be skim-machined if required.

Summarising these advantages, a more efficient structure can be designed, nearer to the ideal, since configurations can be used which could not be manufactured at reasonable cost by conventional methods.

Manufacture and Production

(a) Metal can be removed after a forming operation, thus preventing the formation of flats on a contour between integral stiffeners. This advantage solves a production problem which has been urgent ever since the invention of the large skin-milling machine.

(b) Semi-skilled workmen operate the process using low capital cost equipment, thus releasing skilled operators and expensive machines for other work. It is easier to maintain close thickness tolerances by contour etching than by any other method of metal removal.

(c) Curved lines can be produced as easily as straight lines: different planform radii may be contoured simultaneously on the same component. These facilities help the Planning Engineer.

(d) Sheet material can be etched on both sides simultaneously, thus preventing the warping which would take place if the sheet were machined first on one side and then on the other.

(e) Tapering of skins or longerons becomes a simple operation using contour etching.

(f) Integral ducting can be produced by making the skin in two halves and mating the two etched grooves vis-vis. Such ducting need not be straight, and the only conventional method of producing it at reasonable cost is by casting.

Economic Advantages

Many components can now be manufactured which are outside the scope of conventional methods, or which, whilst they could be produced conventionally will be made more quickly and at lower cost by contour etching.

The time taken to produce a component is reduced (Refs. 1, 3 and 5) and so the Direct Labour Cost and also the Job Overhead are reduced.

The labour rates are lower, and plant with a capacity equal to that of one machine is cheaper.

Numerous small components can be etched simultaneously. These may either be nested by using a number of carriers, or they may all be delineated on several large standard size sheets, which reduces handling costs, utilises the bath better, reduces the number of carriers required and saves material by reducing offcut allowance and almost eliminating holding allowance.

Using convenient integral construction, the number of parts per aircraft can be reduced. Time is saved on assembly and detail tool design. The overall tooling cost and also the cost of paperwork and administration are reduced.

3. Review of Existing Processes

The basic sequence of Contour Etching procedure is :

1. Clean
2. Mask
3. Etch
4. Neutralise and wash
5. Strip

Two masking techniques are employed - direct positive and negative/positive.

Direct Positive. The component is sprayed all over with the 'resist' i.e. the etchant-proof coating. The, after cutting round a thin template with a scalpel, the resist is peeled off those areas which are to be etched and the part is ready for etching.

Negative Positive. The component is sprayed all over with a strippable coating. After cutting as before, the strippable coating is peeled off those areas which are to be protected. These areas are then sprayed with the resist, the remaining strippable coating is peeled off and the part is ready for etching.

The former method is obviously the quicker, but until recently a resist with a suitable ratio of tensile strength to peel strength was not available in Britain.

The operation of each existing commercial process has been compared briefly in tabular form, Table 1. In so far as it has been possible to obtain information, the American processes appear to be the best available for contour etching. Unfortunately, the complete information is not available, despite the adequate patent coverage which has been obtained. The author was fortunate in being given some idea of the composition of the American etchants.

As regards the process itself, the greatest single advantage which the American 'Chem-Mill' has over the British processes, lies in the method of applying the mask. The 'direct-positive' technique not only saves the cost of a strippable coating but also the labour and time spent in applying it. Moreover, the Neoprene Resist requires only one coat and takes only one hour to cure as compared with the British Cellon Resist which, counting the undercoats, requires four coats and three hours stoving. The Epoxy resin Resist is somewhat better, requiring only two coats and forty five minutes curing.

A further advantage of direct-positive masks is that, although it is claimed that sharp edges can be achieved with the other type, there is always the danger of lifting or chipping at the edge when the strippable coating is removed. This risk is completely obviated when the removed portion is severed from the remainder.

Considering next the etching techniques, some plants circulate etchants through the centrifuge almost continuously. An intermittent process requires that etching must cease approximately once a fortnight for the etching bath to be emptied and filled again with regenerated solution, which will be cold and may take several hours to reach the process temperature. This operation is time consuming and may occur at a very inconvenient moment. The former method is preferable.

It is interesting to note the different ways which are used to ensure that the component is etched to the correct depth. Company A have incurred extra capital expense in automatic control. Company B uses either a control specimen (for accurate work) or an approximate time calculation based on a varying rate of attack. Company C has sacrificed the rate of attack by lowering it to constancy, thus allowing a more accurate time calculation. Of the three methods, that used by Company A will be the cheapest in the long run, and the most accurate, but for a plant to satisfy the limited requirements at present felt in Great Britain, it would not be economical. Development would stimulate the use of contour etching, and is to be encouraged.

The process which produces the best surface finish is that developed by Company B. A good surface finish should undoubtedly contribute to an increased fatigue life and it should be pointed out that this process makes a specific attempt to maintain and improve fatigue life.

The Company C approach is, however, extremely practical. There are many instances when surface finish and fatigue resistance are not important, or where contour etching could relieve the load on a machine shop by 'roughing out' a batch of components.

The surface finish of components by Company A is quoted as 35 to 60 microinches centre line average (C. L. A.). This range is fairly wide, and in the absence of fuller information must be assumed to cover all the finishes obtained on the different etched allows. It is unlikely that such variation would be found between two components of the same material.

In the final operation of de-masking, Company A has further advantages. With the neoprene or vinyl masks, the 'stripper' is sufficiently cheap and non-toxic to allow application from a bucket and using a mop. Large components may thus be laid on a concrete floor whilst the operator, wearing waders, walks round swilling them liberally with the 'stripper'. Not only would this be too expensive using a commerical paint stripper - it would be dangerous.

Some manufacturers etch components on both sides. The advantages of this technique have already been described, and Company B follows this practice. The comprehensive fatigue tests which the firm considers necessary have been performed. An investigation on this subject in the case of clad material is described in this paper.

The comments have, so far, been confined to the pocket-producing processes. Two others, Company A and Company D, methods for perforating foil are in Appendix 1 and Table I. These methods fall within the definition of contour etching, and again the Company E appears to be the better, if only for the variety of materials which can be etched.

The most useful development would be an improvement in the masking technique. It could be effected if a light-sensitive coating which was also resistant to the etchant could be found. Such a coating would be useful in all branches of Contour-etching.

To summarise, the most pressing needs of British Industry in the contour etching field are :

1. A direct-positive masking technique
2. More automation
3. A less restricted approval, which can only be achieved by (4)
4. A scientific investigation of any effects on mechanical properties
5. More experience with steels and allows of magnesium and of titanium.

4. Present Work

Because this process is new and so attractive, much development work is being carried out on both sides of the Atlantic but little detail of materials or procedure has been revealed. An investigation has been made to explore suitable materials for etching and masking, and to develop the more attractive direct positive technique.

Work in a new field, on clad aluminium alloy to DTD 546, has been done and the effect on tensile and fatigue properties explored. An attempt has been made to determine the cause of the reduction in fatigue life subsequent to contour etching reported by some workers (Ref. 3).

4.1. Etching - Procedure and Evaluation of Etchant

To assist in the full understanding of the choice of tests, the following summary of experience up to the beginning of the reported programme is given.

It was known that:

(a) Alkaline etchants are better than acidic etchants for alloys of aluminium

(b) The common wetting and inhibiting additives give no advantage

(c) A suitable caustic concentration lay between 10% and 30%. (At that time, it was not known that "A" operates on a basic 7% NaOH etchant).

It was decided to determine, for the three etchants 10%, 15% and 20% NaOH (by weight), the relationship between :

(a) Bath temperature and rate of attack

(b) Bath temperature and surface finish

(c) Surface finish and depth of etch

(d) Rate of attach and Al content of etchant

Procedure

The basic tests are described below. Further results were obtained from sundry specimens used in other parts of the report.

To determine the rate-of-attack curves (Figs. 1 and 2) baths were made up to 10%, 15% and 20% Sodium Hydroxide. Each etchant was used over a range of temperature from 15°C to 100°C to attack carefully weighed and measured specimens of DTD 546B, using a fresh bath at each temperature. The specimens were removed, de-smutted, dried, weighed and measured at 15 minute intervals. The C.L.A. value of surface finish was measured by Talysurf.

The effect of depth of etching on surface finish (Fig. 3) was determined almost entirely from sundry tensile and fatigue test specimens.

To determine what is colloquially known as 'The Bath Exhaustion Rate' (Fig. 4) 1600 cc's baths were made up to 10% and 15% Sodium Hydroxide respectively and maintained at 80°C. Weighed and measured specimens were immersed in these baths for 15 minute periods until the Rate of Attack fell below .036" per hour.

Discussion

Examination of Fig. 1 reveals an interesting phenomenon. Where the rate of attack is low, the variation across the four fifteen minute periods is irregular, but with a tendency to increase. Where the rate of attack is high, it declines with a rapidity inversely proportional to the sodium hydroxide concentration. Where the rate of attack is medium, the variation across the periods is slight.

The rate of attack on pure aluminium is lower than on the core material, partly due to the presence of components in the core material which do not react with sodium hydroxide, but fall out as the surrounding aluminium is removed. Thus, if the rate of attack is so low that the cladding has not been penetrated in the first or second period, a rise will occur in that period in which penetration occurs, and a further rise in the next period, the first in which all the metal removed was core material.

On the other hand, if the rate of attack is high, the cladding will have a small percentage effect and, in the small baths used, a second factor, etchant exhaustion, will take over. It will later be shown that its effect is roughly inversely proportional to the sodium hydroxide concentration. Finally, with a medium rate of attack, a balance will be struck between these two effects. It was because of this phenomenon that the average rate of attack was weighted in favour of the rate most likely for a useful depth of etching in a reasonable sized bath. Fig. 2 indicates the relationship between bath temperature and rate of attack for the three etchants evaluated. The relationship is of hyperbolic form and agrees with those shown in Refs. 3 and 4.

The determined '10% curve' falls away from the expected curve (shown dotted and later confirmed by a check test at 87^{o}C) at high temperatures, because, with the high associated rate of attack, the 10% bath exhausts more quickly than do the others. This effect is, of course, emphasised by the laboratory scale of the experiments, but is, nevertheless an illustration of one of the disadvantages of weak solutions.

Fig. 5 relates bath temperature to surface finish, the curve for the 15% etchant will be discussed first. This curve shows a deterioration in surface finish as the bath temperature is increased, up to approximately 60^{o}C, after which the surface improves up to 85^{o}C. Above this temperature the surface rapidly deteriorates. The reason for this somewhat strange relationship is believed to be that, in this type of test, two factors affect the surface finish. At low temperatures, the reaction is not very violent and the cuprous and cupric oxide smuts which are formed adhere to the surface. They thus form a semi-permeable 'mask' which allows local attack and hence induces a poor surface finish.

At high temperatures, the rate of attack is also high, and any slight inhomogeneities in the material being etched will again initiate local attack. In confirmation of this theory, it has been found that after etching at 60°C, the smut is indeed thick and difficult to remove, even in nitric acid. It is obvious, therefore, that at 85°C the reaction is sufficiently violent to clean off the smut as it is formed, thus giving a uniform rate of attack, but not sufficiently violent to produce a rough finish per se.

A similar curve has been obtained for the 10% etchant. At low temperatures, the smutting effect is not so severe, because the rate of attack is lower than in 15% NaOH, which means that less oxide is formed. Above 80° however, the 10% etchant gives a worse surface finish than does the 15% etchant. This phenomenon was noticed by M. C. Sanz (Ref. 1). It will be realised that the determined curve again shows the effect of bath exhaustion. It is interesting to note that in this case, the exhausted bath gives a better finish than would be expected in a fresh bath (see confirmed extrapolation). This effect is due to the lowering in rate of attack relative to the expected value and is not a merit of the exhausted bath. The findings in no way conflict with those of other workers (Refs. 3 and 4), who have shown that at 80°C exhausted baths give poorer surface finishes, because at that temperature, the lowering in rate of attack is not sufficient to compensate for any deleterious effects of exhaustion itself.

The curve applying to a 20% etchant is the exception which proves the rule. In this case, even at 60°C, the reaction is sufficiently violent to remove the smuts, and the surface finish is good, getting progressively worse as the temperature (and rate of attack) increase. Unfortunately, above 90°C the reaction is uncontrollable and, in a small bath, dangerous. Large amounts of heat are generated, which makes temperature control difficult, and large volumes of hydrogen are evolved, which aerate the etchant to such an extent that the rate of attack is unpredictable. A probable extrapolation to the curve is shown dotted.

Comparing Fig. 5 with the curve given in Fig. 3 there appears to be little resemblance, at first sight. It must be remembered, however, that the curves in Fig. 3 range only from 60°C to 90°C, and difference in bath size will have an appreciable effect within the critical range.

It was thought that surface finish might be dependent solely upon rate of attack, regardless of the temperature and concentration conditions under which the rate was obtained. In this case, the cross plot of surface finish vs. rate of attack shown in Fig. 6 would have been a smooth curve, with all the points for all the concentrations lying on it. The three distinct curves actually obtained indicate that no such relationship exists.

An interesting relationship has been derived between the depth of etching and the surface finish (Fig. 3). The most likely explanation of its form is that surface finish is dependent upon the degree of work hardening of the etched material, deteriorating in areas which have been least worked. The worst surface finish is produced at the centre of the specimen, where there is least work hardening. It will be appreciated that if a specimen is etched deeper than 50% of its initial thickness, it will have the poorest finish as the 50% mark is reached, but, subsequently, material being etched will have increasing degrees of work hardening and deterioration will not continue. The finish will, in fact, improve as shown, because attack takes place on the flanks of any irregularity as well as on its peak. This tends to level the surface so that Fig. 3 becomes asymptotic at about 40 micro inches. The dotted line in Fig. 3 shows that the surface finish is worse for small baths than for large. This is probably due to local exhaustion.

In determining the rate of bath exhaustion, it was found (Fig. 4) that a 15% solution of sodium hydroxide at 80°C will attack at the rate of .060"/hr. until the aluminium content has risen to 17.5 gms/litre (1.52% by weight). The rate then falls to .040"/hr until the etchant contains 35 gms/litre (3.04%) of Al. Similar figures for a 10% solution are 15 gms/litre (1.36%) and 30 gms/litre (2.72%). It was decided on economic considerations that .040"/hr would be the lowest allowable rate of attack.

<u>NOTE</u> In this context, the phrase 'Weight of Dissolved Aluminium' is used. Strictly, all this weight is not aluminium, being partly composed of the alloying elements but as their percentages are small, they have not been separated.

The reason for this inhibition of the rate of attack is thought to be that the reaction slows down in the presence of aluminium oxide. Removal of the oxide should recover the original rate of attack. This is the purpose of the 'seeding and regeneration' procedure carried out by Company B and of the continuous centrifuging carried out elsewhere.

During the present investigation, when the rate of attack dropped below .040"/hr at 80°C all etching was stopped and the bath was left to settle for a week. The top liquor was then syphoned off into another tank and the sludge was scraped and washed out of the etching tank. On replacing the top liquor and making up to 15 gallons with 15% NaOH solution, the rate of attack was completely resotred. As a further test of the efficacy of this method, the etchant was analysed at the end of the investigation (after 8 months use). The top liquor was found to be 15.4% NaOH by weight and to contain 17.5 gms/litre of dissolved aluminium. As would be expected from Fig. 4 the etch rate was, at that time, slightly less than .060"/hr.

Another parameter was noticed during these tests. In the experimental plant, an etching area of 20 sq. in. /gallon gave sufficient exothermic heat to maintain the temperature at approximately 80°C. If a greater area/gallon was etched, the temperature could only be kept down to 80°C by cooling. This parameter would be increased for larger baths having a greater cooling area (c.f. 25 sq. in. /gallon given in Ref. 3).

4.2. Masking material - Properties, Procedure and Evaluation

The requirements of a good masking material are :-

(a) It must completely resist the etchant at temperatures up to 90°C

(b) Any stoving or drying treatment must not require a temperature above 135°C. (A. P. 970 states that, apart from the specified heat treatments, aluminium alloys may not be heated above this temperature).

(c) It must either be sufficiently pliable to fold up and back, or sufficiently brittle to break off, as under-cutting of the mask line takes place (Ref. 5).

(d) It must adhere sufficiently strongly not to peel off under the violence of the reaction.

(e) It must be sufficiently durable and elastic to allow forming the component after masking.

(f) It must be possible to delineate a sharp, accurate edge with it.

(g) It must be reasonably clean and easy to apply.

(h) It must be easily strippable after etching

(i) It must be reasonably cheap.

Two masking materials are here evaluated,selected as being the most promising from the range shown in Table 2.

The masking materials evaluated during the present work were :-

(a) Cellon 4SL. 083 epoxy paint
(b) British Paints PR. 500 liquid neoprene.

(a) Cellon 4SL. 083 is the masking material used by Company B and satisfactory results have already been achieved. The purpose of further investigation was to determine what troubles might be likely to arise in its use, what restrictions would have to be imposed and how far the existing restrictions might be relaxed.

(b) PR. 500 is a black neoprene-based coating, supplied by British Paints Ltd. of Newcastle. It is normally used as a protective coating for plant or components which may be subject to a corrosive atmosphere. Because it is flexible and resistant to alkaline attack it possesses the basic requirements of a masking material. It later became apparent that it could be used as a 'direct positive' masking material, and this was the main reason for persisting with the evaluation after the poor initial results.

Procedure

(a) Cellon 4SL. 083

The accepted method of application and the approved alternative for producing a chamfered instead of a radiussed fillet were tried. They are described in Refs. 3 and 6.

The following variations to the accepted techniques were tried :-

1. Only one undercoat of PR. 30B
2. Precleaned by degreasing in trichlorethylene only
3. No anodising
4. Reduced stoving time for final resist
5. Only one coat of final resist.

The following proprietary stripping agents were tried :-

1. Cellon MH. 54120
2. Ardrox 666
3. Stripalene 713

(b) British Paints PR. 500

The normal method of applying this coating is by brushing. On enquiry to the Suppliers, it was found that the thick treacly liquid as received could be thinned to a sprayable consistency by the addition of British Paints Thinners T. 146, but, as previous experience had shown the difficulty in overcoming flocculation when spraying other elastomers, the prospect was not relished.

The following pre-cleaning and application techniques were investigated :-

(a) Degrease in trichlorethylene, brush apply one coat
(b) Degrease in carbon tetrachloride, brush apply one coat
(c) Degrease in acetone, brush apply one coat
(d) Degrease in trichlorethylene and anodise, brush apply one coat
(e) Pre-etch in 15% NaOH, brush apply one coat
(f) Degrease in trichlorethylene and methylated spirits, brush apply one coat
(g) As (f), brush apply two coats
(h) Degrease as in (f), dip apply one coat
(i) Degrease as in (f), dip apply two coats

The following technique for delineating the contour were investigated.

(a) Negative-positive (with Sellotape or Birlon No. 3)
(b) Direct-psoitive (with one or two coats)

The mask was cut by running a ground and honed hacksaw blade along a rule for straight edges and round a radius gauge for circular arcs.

The effectiveness of the mask was determined in terms of the time for which it would resist a 15% sodium hydroxide solution at $80^{\circ}C$. In this test, a coated specimen was immersed in the etchant and examined at half hourly intervals for signs of deterioration. From the same specimen it was determined how deep it was possible to etch before the contour edge became jagged.

The durability of the mask was determined by applying one coat, as in 4.2 above, to a specimen of 16 SWG. DTD.610B, $4\frac{1}{2}$" wide x 7" long and air drying for one hour. A contour was then delineated by the direct-positive technique and the specimen was formed as in Plate I to represent say, a leading edge. The forming was carried out by placing a sheet of thick brown paper on both sides of the specimen and rolling it in hand rollers to a 2" radius, leaving the lower 2" flat. The flat end was then inserted in a folding machine and folded up. The smallest radius which could be achieved on this machine was 3/16", at which the mask showed no sign of rupture. The total angle of forming was 180".

If the masking material is applied in the manner which was found to be best (see 6.2) it may be peeled off after etching. The following solvents were also evaluated.

(1) Acetone	(2) Cellulose thinners
(3) Methylated spirits	(4) Trichlorethylene
(5) Toluene	(6) Xylene

Discussion

(a) Cellon 4SL 083

The accepted method of application and the approved alternative were found to be the only methods which gave satisfactory results. In fact it was found that even with the Titanine undercoat, a contour corner radius of less than 0.1" could not be held. The Cellon undercoat did, however, give a tapered fillet inclined at about 30^{o} to the surface, as described in Ref. 3.

Of thre three 'strippers' evaluated, Cellon MH 54120 carely softened the coating in 2 hours; Ardrox 666 removed the mask in about 1 hour; Stripalene 713 removed the mask in about $\frac{1}{2}$ hour.

(b) British Paints PR.500

None of the pre-cleaning techniques described in Sec.4.2 (a) to (e) inclusive gave a satisfactory edge. For the most part, the contour edge was jagged due to peeling of the mask ahead of the etching front. The mechanism of the formation of defective edges is fully explained in the References.

When the surface of the component was finally cleaned with methylated spirits, a good edge was produced. Now, in the preceding tests, the contour had been cut with a knife and the part requiring etching had been scraped off, i.e. using a direct positive technique. This, however, was time consuming since only a small area would peel off at a time. It was at this stage that attention was turned to a 'negative-positive' technique. Sellotape was used for the first negative but on removal it left a very ragged edge, and the mask was removed completely so that a fresh start could be made. At the second attempt, Birlon No. 3 was used, and gave a very good edge to the positive, when removed.

It was still hoped that the application sequence could be modified to allow direct-positive usage, so a specimen finally cleaned in methylated spirits was given two brush coats, air drying for one hour between coats and finally curing for $\frac{1}{2}$ hour at 120°C. When the contour had been cut, it was found that, at last, a coating had been achieved with sufficient adhesion not to peel off in the etchant, but sufficient tensile strength to allow large areas to be peeled off in sheets, by hand. This specimen, shown in Fig. 7 has good sharp contours and a well formed full radius fillet. It was step etched by leaving the centre portion masked until the speimen had been in the bath for $1\frac{3}{4}$ hours, then removing it, peeling off the centre portion and continuing etching for 30 minutes.

The only blemish on this otherwise perfect specimen is that the cladding, having been etched at a slightly slower rate than the core material, projects about .005" at the edge, forming a slight lip on the fillet radius. This feature is not caused by too stiff a masking material as shown in Ref. 5, but is inherent in the deep etching of clad materials. It is, in any case, a very small lip which would be easily removable by running a scraper along the edge.

Reference has already been made to the final specimen shown in Fig. 8. The mask stood up very well to the forming operation and showed no sign of bursting, or lifting from the inner surface. A few slight blemishes were suffered in places where either dust had become embedded in the mask during drying or particles of grit on the rolls had penetrated it.

This difficulty emphasises the need for a dust free masking area and utmost cleanliness, coupled with good Inspection. This need has been remarked upon by other workers, and is not peculiar to the use of PR.500.

Some other information may be gleaned from this test. Just visible in Fig. 8 is the rectangular panel on the flat portion of the specimen. This panel was cut to sharp corners, which have been etched into approxima tely .030" contour radii, far sharper than was obtainable with the Cellon masking material. The integral stiffener down the centre is only 0.1" wide yet the mask shows no sign of lifting. After the first few tests, indications were that PR. 500 would mask the edges effectively and thus do away with the necessity of using tapes along them. It proved unreliable, however, so tapes should still be used. The mask can be peeled off after etching, but a better way is to soften it in any of the last three solvents evaluated; trichlorethylene, toluene or xylene and then wash it off in hot water. The whole demasking operation then takes about 10 minutes, as compared with 30 minutes for Cellon 4SL 083.

It has been seen that two brush coats or one dipped coat will give a mask sufficiently adhesive to give a good edge and sufficiently strong to allow it to be peeled off where required. When a specimen having a single dipped coat on one end and two dipped coats on the other was immersed in the etchant and examined at regular intervals, it was found that the single coat began to blister after about 4 hours, and the double coat broke down after $6\frac{1}{2}$ hours. This effect limits the allowable depth of etching to about 0.2" but is not a serious disadvantage since the majority of normal aircraft work will not require deeper edges.

5. Methods of Mechanical Testing

Since the inception of contour-etching, metallurgists and the controlling bodies of the aircraft industry have been worried about its effect on mechanical properties. It was not until Vickers-Armstrongs (Aircraft) Ltd. published results for (unclad) DTD. 646B in January 1957 (Ref. 3) that anything was known on this side of the Atlantic. Some rather inconclusive data had been published in October 1954, (Ref. 1) and some still less satisfactory data had been published in May 1956 (Ref. 5) but these did little more than create suspicion.

The present investigation is concerned specifically with the effects of contour etching on the mechanical properties of (clad) DTD. 546B in the fully heat treated condition. Concurrently with the Fatigue Testing, and before the final results were known, an attempt was made to determine the reason for the expected loss of fatigue strength discussed in 5.2.2. Finally, an appreciation is given of the present position in so far as alloys other than those tested are concerned.

NOTE: In this context, the word 'etching' refers to contour-etching in 15 gallons of a 15% NaOH solution at 80°C, unless otherwise stated.

5.1. Tensile Properties

Tensile tests were performed at all stages of the investigation so that it could be determined whether any unexpected phenomena could be ascribed to changes in tensile properties. The specimens used were to B.S.S. 3A4 (Tensile specimens for sheet material, 0.5" wide employing a 2" gauge length). All specimens were cut with the direction of final rolling running longitudinally.

In order to determine what effect, if any, the depth of etching had on tensile properties, a series of 8 SWG. sheets of DTD. 546B was etched on both sides from 0.162" thick to .126", .094", .060" and .032" thick. This process involved removing 20%, 40%, 60% and 80% of the original thickness.

The results are given in Table III. Tensile tests on specimens cut from the same sheets as the fatigue test specimens are given on the S/N curves for the specimens which they represent.

Discussion of Effect on Tensile Properties

Referring to Table III relating depth of etch to tensile properties, the first obvious feature is that the U.T.S. and .1% P.S. increase after the first etching. This is wholly attributable to the removal of the weak cladding, and is not peculiar to contour etching. Deeper etching appears to reduce both these values slightly, but the reduction is so slight that it cannot be claimed that there is any correlation between depth of etch and either U.T.S. or .1% P.S.

The 3% elongation also appears to be reduced, but this is simply an example of Barbas' Law, i.e. there is an approximate linear relationship between the % elongation and the square root of the specimen thickness. If the gauge length had, in each case, been taken as $4 \times \sqrt{\text{cross sectional area}}$, the % elongation would have remained constant. This control would, however, have been impracticable, because the gauge lengths would have varied from .506" to 1.138".

The effects of post heat treatment are shown in Table IV. For material etched on one side only, the U.T.S. and 0.1% P.S. increased at the expense of the % Elongation, whilst, in the material etched on both sides, the reverse occurred. The changes are so slight that they will have no effect on fatigue life.

The tensile properties of the materials used in the other fatigue tests give no cause for comment.

5.2. Fatigue Properties

Since chemical attack would appear to roughen the surface and probably make a preferential attack at the grain boundaries, it could be anticipated that stress raisers would be formed and that the fatigue strength would be reduced, perhaps considerably. Other conditions which could produce a similar loss of strength are the removal of a surface skin induced by previous shaping processes, and the diffusion of hydrogen into the surface, producing brittleness. These possibilities made an investigation of the fatigue strength of contour etched material quite urgent.

5.2.1. Testing Machines and Test Pieces

Two machines were used, both of the Avery reverse bend type for sheet. The first was modified by Steen (Ref. 16) and was used for most of the tests. The second machine was modified by the author to take ten test pieces at the same time. For further details the original thesis (Ref. 18) must be consulted. (Fig. 9).

The details of the strip test piece are shown in Fig. 10. The thickness was 0.080". All test pieces were cut with the longitudinal direction, that of the final rolling. Fly milled test pieces were machined so that the cutter centre traversed the centre line of the test piece. Appendix II is devoted to the design, development and use of the fatigue testing machines, so the testing procedure will not be described here.

Tests were performed on specimens which had received the following treatments :-

TEST I

Fly-milled one side only, polished both sides on grades of emery down to 3/0 and finished on an alumina pad.

TEST II. Fly-milled one side only, surfaces left 'as machined' and 'as rolled'.

TEST III. Contour-Etched, one side only.

TEST IV. Contour-Etched both sides.

TEST V. Contour-Etched one side only, post heat treated ½ hr. at 175°C.

TEST VI. Contour-Etched one side only, post heat treated 1 hr. at 175°C.

TEST VII. Contour-Etched one side only, post heat treated 2 hrs. at 175°C.

TEST VIII. Contour-Etched one side only and 'Vacu-Blasted' on that side.

TEST I. This test was intended to be the basic control test. The surface finish of the specimens averaged 5 micro inches C.L.A.

TEST II The average C.L.A. value taken along the direction of final rolling (and of machine traverse) was 70 micro inches. In the transverse direction, the value was 50 micro inches.

TESTS III & IV These specimens had surface finishes with C.L.A. values of 32 and 50 microinches in the longitudinal and transverse directions respectively.

TESTS V, VI, VII and IX These tests were performed to determine whether heat treatment could improve the fatigue life of clad material.

Heat treatment was carried out by attaching a thermocouple to a control specimen and placing it in an air circulation furnace. The temperature control was adjusted until the control specimen was holding a temperature of $175^{\circ}C \pm 2^{\circ}C$. The specimens, one of which was fitted with a second thermocouple, were then placed in the furnace for the prescribed time, after which they were removed and air-cooled.

The temperature of 175°C was chosen because this is the Precipitation Treatment temperature for the material. Although AP. 970 lays down that, in general aluminium alloys should not be heated above 135°C, the DTD. Specification allows precipitation treatment for between 5 and 20 hours, dependent upon the component size and required properties. The higher temperature would, of course, assist in the extraction of any absorbed hydrogen. It soon became apparent that no improvement had been achieved, so the tests were stopped before the curves had been fully 'established' (see definition in A.S.T.M. Manual on Fatigue Testing 1949). Test VII was actually run in two different series - the first series failed to produce a single specimen which remained unbroken after 10^7 reversals, even at stresses as low as + 5 t/sq. in.

TEST VIII 'Vacu-Blast' is a trade name for a method of grit-blasting using a portable gun. The grit, in this case 'Blastyte No. 6' (Al_2O_3), is ejected through a central nozzle and sucked back through a concentric annulus sealed from the atmosphere by a flexible brush, similar to a vacuum cleaner. The makers of the equipment, Vacu-Blast Ltd., of Slough, treated a 14" x 19" etched sheet, using a Vacu-Blast 35 J Mark II model gun, with which the speed of the operation is 60 sq.ft./hr. using an air pressure of 90 lb/sq.in. The surface finishes obtained before and after blasting had C.L.A. values of 50 and 44 microinches respectively.

The important results of fatigue tests are shown graphically in Figures 11 to 15.

The fatigue endurances of DTD.546B for a life of 10^7 reversals are:

Machined one side and polished	$\pm$ 6.55 T/sq.in.
Machined one side only	$\pm$ 6.10 T/sq.in.
Contour Etched one side only	$\pm$ 5.58 T/sq.in.
Contour Etched one side and 'Vacu-Blasted"	$\pm$ 6.16 T/sq.in.
Contour Etched both sides	$\pm$ 8.80 T/sq.in.

(The other tests were stopped when it became apparent that they showed no improvement. Fig. 16).

Taper sections were taken from the following fatigue specimens:

(a) Unfatigued, showing clad side.
(b) Unfatigued, showing etched side.
(c) After failure, showing clad side.
(d) After failure, showing etched side.
(e) Fatigued but unfractured, showing clad side.

The method of taking specimens and their positions on the test specimen are shown in Fig. 17. All specimens in this section were contour etched on one side only. Photographs of the taper sections are shown in Figs. 18, 19 and 20. The correspondence with both the 'as machined' and 'as etched' and 'Vacu-Blasted' curves is very close, bearing in mind the the inevitable scatter on S-N curves.

Fig. 21 shows the effect of post heat treatment. If anything, the fatigue properties have deteriorated, and no satisfactory explanation has been found from examination of the tensile properties. The problem is worthy of special investigation, but, for the purpose of the present work, is of academic interest only, since post heat treatment is not envisaged.

The possible reasons why contour etching alone reduces fatigue endurance (para. 6), must be discussed. An unexpected phenomenon was noticed during the tests. Of the 110 clad-one-side specimens which were broken, in every one, so far as could be determined by examination of the fracture and observation of the specimen at the moment of failure, the fatigue cracks started from the clad side. This tendency was so decided that when a series of asymmetrically strained specimens was run, the asymmetry had to be increased to give a nominal stress of 10 T/sq.in. $\pm$ 10 T/sq.in. (clad side in compression) before a crack could be initiated from the other side. The vulnerability of cladding has been noted by P. L. Teed, who records 11 endurances, at a life of 20×10^7 reversals of 8.3 T/sq.in. and 5.8 T/sq.in. for unclad and clad materials respectively.

In confirmation that the cladding was, in fact, cracking first, the taper sections exhibited in Figs. 19 and 20 were examined. Fig. 22 confirms that although the etched surface may become slightly rougher after fatigue testing, it does not crack, even at failure of the specimen.

The clad surface after fatigue testing is shown on the taper sections in Figs. 19 and 20. The surface had been noticed to take on an orange peel effect and this is seen to be formed of major disruptions, which initiate cracks irradiating the entire thickness of the cladding and penetrating the interface to continue in the core material. It will be realised that where a crack appears to be discontinuous, the join crack has merely disappeared either above or below the section face.

It has been conclusively established that if the cladding is removed from one side of a specimen, fatigue failure will be initiated at the remaining clad surface. Why then, should different methods of removal, machining and etching, give the material different endurances ? Experimental work has shown (Refs. 11, 12 and 13) that rolling, shot peening and vapour blasting improve fatigue life as a direct result of the hard skin they induce. Contour etching obviously removes any such skin without replacing it, even partially, as does machining. See Fig. 22.

5.2.2. Discussion of the Effect on Fatigue Properties

Examining the scatter bands exhibited in Figs. 11 to 15 one of the first of the features which are noticed is that the bands are narrower if the material is etched, than if it is machined. The reason for this is that the surface, apart from having a better C.L.A. value than an average machined surface, is broken up into a series of small irregularities rather than being composed of fewer large notches. Fig. 18 illustrates this phenomenon, and it can be seen that the etching pits have rounded bottoms on the micro scale as well as on the macro scale. Attack is not intergranular and proceeds with random uniformity (Refs. 4 and 6). These factors reduce the effect of stress raisers rather than forming them.

The importance of this point is that the 'probable endurance' as derived statistically from the scatter will be closer to the experimental figure for etched material than it would be for machined material. Even if the design stress had to be lowered as a result of these tests, the experimental values could be used with more confidence.

That it need not be lowered is shown in Figs. 15 and 23. If a component is 'Vacu-Blasted' after etching, the fatigue endurance is increased beyond, even, the endurance of the machined material. Previous workers have noticed (Ref. 5) that vapour-blasting improves endurance, but this milder treatment has not proved adequate. Grit blasting by 'Vacu-Blast' work hardens the surface to a greater extent (in addition to improving the finish), and, in the form used in these tests, is very easy to operate on a production scale, even on complicated components.

Using the polished specimens tested in Test I as a basis of comparison gives rather a pessimistic idea of the loss of fatigue properties due to contour etching alone, because they represent an ideal, unobtainable in production work. It is fairer to compare Test III with Test II. Comparison cannot be made with the 'as received' material because it is clad on both sides, and is twice as thick. A curve from the Aluminium Research Laboratories Bulletin No. 1 (March 1952) which applies to 14 SWG. (.080") DTD.546 is reproduced in Fig. 24.

That machining does, in fact, raise the surface hardness relative to the core hardness is shown by Fig. 22. The hardness at various distances from the surface was measured using the G.K.N. Microhardness Tester on the cross sections of two sheets of DTD.746, one of which had been machined, whilst the other was etched. Machining induces an increased hardness within 70 microns of the surface, whereas the etched sheet shows a normal scatter at all depths. The etched sheet was obviously slightly harder overall, but this does not affect the general conclusion.

In this context, a further point should be considered. If a rolled material is contour etched on one side only, the work hardening will be removed from that side, but will remain at the other. This process has the effect of moving the neutral axis of the cross section, so that if the specimen is subjected to a symmetrical, constant strain, reverse bend fatigue test, it will, in fact, suffer an asymmetrical stress. Now it is well known that asymmetrical stressing is more dangerous than symmetrical stressing, and it may well be that low test results can be accounted for in this manner. This phenomenon would be especially noticeable in single sided clad specimens, but would also apply to unclad specimens such as DTD.646.

It can now be explained why grit-blasting the etched surface improves the endurance to equal that of the machined-one-side specimens.

One aircraft firm has repeatedly warned against hydrogen embrittlement. In support of this warning is the increase in fatigue life claimed when specimens are heat treated for 2 hours at 135°C after etching. This is the only evidence which the firm has found. Enquiry from various sources has failed to produce any corroborative experience of aluminium alloys suffering in this way, beyond that of Moreau and Chaudron (Ref. 14). It was later shown that their hydrogen extraction apparatus was faulty (Ref. 15). Eborall and Ransley (Ref. 7) go so far as to state categorically :

> "The solid solubility of hydrogen in aluminium and its alloys under atmospheric pressure is clearly very small".

This conclusion followed a demonstration that, after maintaining an aluminium alloy at 100°C surrounded by hydrogen at atmospheric pressure, the concentration of hydrogen is only 1% of the surface concentration within .002" of the surface after one month. Since the rate of penetration of molecular hydrogen is therefore lower than the rate of metal removal by contour etching, there can be no penetration into the final surface.

Apart from the discredited Moreau and Chaudron experiment the above evidence relates specifically to molecular hydrogen. There remains the possibility that embrittlement might be due to the action of nascent hydrogen as it is formed at the surface. The diffusivity of nascent hydrogen into aluminium alloys has not yet been satisfactorily determined, owing to the difficulty of either maintaining the hydrogen in its nascent form during diffusion, or determining at what point it associates to H_2.

Experiments by Duflot (Ref. 17) in which he consciously attempted to charge his specimens with nascent hydrogen by cathodic action, show that the rougher the surface finish, the less hydrogen penetration will occur. He concludes : "... le polissage electrolytique donne l'etat de surface le plus favourable a la penetration de l'hydrogene : vraisemblablement parce qu'il elimine ou diminue les centres ou se fait la recombinaison $H + H \rightarrow H_2$". (Electrolytic polishing gives the most favourable surface condition for the penetration of hydrogen, probably because it eliminates or reduces the centres where the recombination $H + H \rightarrow H_2$ occurs).

Now, relative to electrolytic polishing, contour etching gives a rough surface, ideal for the formation of 'centres de recombinaison'. Moreover, as already pointed out, unless the rate of penetration of nascent hydrogen is greater than .001"/min., hydrogen is greater than .001"/min., hydrogenation is impossible.

A specimen of 8 SWG. DTD.546B, as received, was sent to the British Non-Ferrous Metal Research Association's Headquarters and Laboratory in Euston Square, London. Here 040" was machined off both sides and a determination of the hydrogen content of the core was made using the method described in Ref. 7. The hydrogen content of a similar sheet which had been etched in fresh 15% NaOH solution at 80°C was also measured.

The results of the hydrogen determination were as follows:-

A.	'As received' sheet, machine to .080" thick	0.35	cc's H_2/100 gms
		0.35(5)	" " "
B.	Etched sheet	0.33	" " "
		0.35(5)	" " "

There is no significant change in the hydrogen content so there can be no significant effect on the fatigue strength due to hydrogen embrittlement.

Finally, an appreciation of the present position as regards the etching of alloys other than those tested is given.

No explanation or confirmation of the improvement after post heat treatment experienced by Vickers Armstrongs (Aircraft) Ltd. has been found. It has, however, been established that no hydrogen is absorbed, so hydrogen embrittlement cannot occur. Fig. 24 compares a curve relating to DTD.546 'as received' with the present work and the approved work. The three are comparable, because when the cladding is removed from DTD.546B, the remaining material should be to specification DTD.646B. It can be seen that contour etching on both sides, without post heat treatment produces no deleterious effect on the endurance for a life of 10^7 reversals, although the form of the S-N curve is unusual (para. 5.2.1.). The 'improved' etching technique shows a drop of 40% when etching on one side only. A partial explanation of this is that, since one work hardened surface was left, the neutral axis was de-centralised and the endurance for symmetrical straining was correspondingly lower. It must also be remembered that these results were obtained from material of a variety of batches, whereas the present work was confined to one batch. A third factor will be the difference between the two specimen shapes and loading methods used.

Whilst it is not possible, without a thorough familiarity of the materials and tooling methods used, to give a firm opinion, there is every indication that contour etching need not be restricted to one side of a material to Spec. DTD.646.

The form of the curve obtained in the present investigation for material etched on both sides is not typical of aluminium alloys. It exhibits characteristics indicating a sharper definition of a 'fatigue limit' normally associated with, say, steels. This could be the result of the stress relieving properties of etched surfaces, here affecting both sides of the specimen.

6. Conclusions

6.1. Etching

The best of the etchants evaluated is a 15% NaOH solution operating at 85°C. This gives a rate of attack of .077"/hr. and a surface finish (in a small bath) of 52 micro inches.

For production usage, the operating temperature should be reduced to 80°C because (a) the slightly lower rate of attack (.060"/hr.) will allow either more accurate work or less stringent control for the same accuracy and (b) a wider temperature latitude may be allowed without either the surface finish or the rate of attack varying so much as at 85°C.

In a large bath operating at 80°C, the surface finish will never exceed 52 micro inches and if less than 30% or more than 60% of the initial thickness is removed, it should not exceed 45 micro inches and may be better.

After an initial period of constant rate of attack at .060"/hr. the etchant will exhaust at the rate of .0012"/hr. per grm/litre of dissolved aluminium.

The rate of attack can be recovered by centrifuging the etchant or by decanting it.

The area of material being etched should not exceed 20 sq. ins/ gallon of etchant.

The 10% NaOH etchant was rejected because

(a) The rate of attack is relatively low
(b) If the temperature is raised in an attempt to increase the rate of attack to .060"/hr., the surface finish deteriorates to 80 micro inches.
(c) The bath exhausts more rapidly and would require more frequent regeneration than does the 15% solution.

The 20% NaOH etchant was rejected because

(a) The reaction is too violent, making it difficult to control and dangerous
(b) If the bath temperature is reduced to, say, 60°C to reduce the violence of the reaction, the rate of attack falls to .025"/hr.

6.2. Masking materials and procedure

Cellon 4SL 083 is a useful masking material, particularly for deep etches, but it is essential that the conditions laid down in Ref. 3 be maintained. No relaxation of them has given satisfactory results. Its major disadvantage is the time it takes to apply it.

British Paints, PR.500 is an excellent masking material for etches no deeper than 0.2". One dipped coat is adequate, but in production it may be necessary to use two coats if conditions are not perfect. Sharper contour corners may be produced using PR.500 than by using 4SL 083. When forming after masking, the rolls should be wiped clean and the component sheathed in stiff paper.

PR.500 and 4SL 083 both require augmentation by protective tapes along the edges of the component.

Methylated Spirits has been found to be the best pre-cleaning solvent, but further tests are required to discover a cleaner more suitable for production work. The importance of a dust free atmosphere in the masking area cannot be stressed too strongly.

PR. 500 is resistant to the etchant after 1 hour air drying, but is still tacky after this time. The mask can be toughened by boiling in water for about $\frac{1}{2}$ hour. For production work, it should be cured for 2 hours at 100°C. This compares with a total of 3 hours drying time, plus five spraying operations when using Cellon 4SL 083. The greatest single advantage of PR. 500 is that it can be dip applied using the 'direct-positive' technqiue, thus saving the time required for the intermediate operations.

6.3. Effect on Mechanical Properties

Contour etching has no effect whatsoever on the tensile properties of aluminium alloys.

Etching DTD. 546B on one side lowers the fatigue endurance for a life of 10^{7} reversals by 10% more than does machining. The endurance may be recovered by 'Vacu-Blast' treatment. However the material is removed, fatigue cracking will be initiated from the remaining clad side, due to decentralisation of the neutral axis and the vulnerability of pure aluminium to fatigue failure.

If DTD. 546B is etched on both sides, the endurance will be 57% higher than if it is etched on one side only. This is due to the removal of the cladding. Material etched on both sides is particularly resistant to fatigue at high cyclic stresses with zero mean stressing. There are indications that unclad material will not be harmed by etching on both sides.

Contour etching does not cause hydrogen embrittlement. The current tests show no improvement in fatigue endurance accruing from post heat treatment at 175°C.

The fatigue test results of contour etched material exhibit less scatter than do those of machined or polished material since the etched surface has a stress relieving effect which counteracts the stress raising effect of any extraneous notches.

Some of the specimens were etched six months before testing and protected only by a dip in de-watering oil after etching. No corrosion effect could be discerned. Further, no corrosion or intergranular attack is visible in Fig. 18. These results confirm the experience of other workers.

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TABLE I

COMPARATIVE SUMMARY OF EXISTING PROCESSES

Name	Developed by	Etchant	Masking Material	Mask Type	Rate of Attack	Surface Finish	Special adv. or disadv.
'Chem-mill'	North American Aviation Inc.	7% NaOH + Additives	Vinyl Resin or Neoprene	D/P	.060"/hr.	35-60μ"	Wide range of alloys Electronic control
'Chemi-cut'	Chance-Vought Aircraft Inc.	Unknown	Resistant Ink	N/P	.060"/hr.	-	Perforates .006" stock wide range of alloys
Contour Etching	Vickers-Armstrong (Aircraft) Co. Ltd.	15% NaOH (Regenerated)	Epoxy-Vinyl Paint (Cellon 4SL-083)	N/P	.060 to .040"/hr.	30-40μ"	Radiused or chamfered edge, single sided etching only
Contour Etching	Saunders-Roe Ltd.	12% NaOH (Seeded)	Epoxy Resin or Bitumastic Paint	N/P	.045"/hr.	$\ngtr 50\mu''$ or $> 50\mu''$	Constant rate of attack
Chemical Etching	Technograph Electronic Products Ltd.	Ferric Chloride	Bitumastic Powder	N/P	.024"/hr.	-	Perforates .005" Ferry Foil

TABLE II

SUMMARY OF REJECTED MASKING MATERIAL

Type	Material or Method	Reason for Rejection
d/p	Mechanical Masks (Rubber faced steel templates)	Tend to deteriorate and scrap several components before replacement. Cumbersome in application.
n/p	Conventional aircraft finishes. Cellon, Dockers and Titanine	Lack of properties (a), (c) and (d).
n/p	Dockers Resistant Paint	Lack of property (c).
d/p	Photo and Litho Printing	Resistant ink not available in U.K. This method is used in the U.S.A.
n/p	Silk Screen Printing	Lack of properties (f), (g) and (i).
n/p	Plating (Cu)	Lack of properties (g), (h) and (i).
n/p	Araldite (985E and type 1)	Lack of properties (b), (c) and (g)
n/p	New Bakelite Resins (Polyester and Epoxy) Resorcinol Resins	Lack of properties (b), (c), (g) and (f)
Materials for "negative" masks	"Sellotape" "Scotch Tape", etc.	Lack of property (f), (i.e. final resist is torn on removal of tape).
	Thin gauge steel templates	Lack of property (f) (except when pulled down by magnetic chuck). Then rejected for lack of properties (g) and (i).
	Paint inhibitors	Lack of property (g), (involves "positive-negative-positive" technique). Partial lack of property (f)

TABLE III

EFFECT OF DEPTH OF ETCH ON TENSILE PROPS. AND SURFACE FINISH OF D.T.D. 546

SPECIMEN No.		THICKNESS 't' ins.	U.T.S. T/□"	.1% P.S. T/□"	% EL. on 2" G.L.	SURFACE C.L.A. μins.	AV. U.T.S.	AV. .1% P.S.
1	A	.1625	30.0	24.6	11.3	5	29.8	26.2
	B	.1620	29.8	27.4	13.0	5.5		
	C	.1620	29.5	26.5	7.5*	5		
2	A	.1255	33.1	29.2	10.0	42	32.8	29.5
	B	.1260	32.7	29.8	10.0	43		
	C	.1262	32.5	29.4	7.0	42		
3	A	.0940	33.0	29.1	10.0	46	33.0	29.9
	B	.0940	33.7	29.8	10.5	46		
	C	.0960	32.4	30.0	10.0	46		
4	A	.0620	32.6	29.8	7.5	59	32.7	29.0
	B	.0606	32.4	28.4	10.0	59		
	C	.0600	33.1	28.3	8.0	60		
5	A	.0324	32.5	30.2	5.0	55	32.1	29.8
	B	.0325	31.7	29.0	5.0	48		
	C	.0320	32.2	30.2	4.0	53		

* BROKE AT GAUGE MARK

TABLE IV

EFFECT OF POST HEAT TREATMENT

DESCRIPTION	SPEC. NO.	U.T.S. T/ "	.1% P.S. T/ "	% EL. 2"G.L.	AV. U.T.S.	AV. .1%P.S.	AV. % EL.
One side clad as received One side contour etched No post ht. treatment	1 2 3*	31.0 29.0 30.5	24.4 21.3 28.1	10.0 8.0 17.5	30.2	22.8	11.7
Contour etched both sides No post ht. treatment	4 5	34.1 33.3	30.7 29.8	9.5 9.0	33.7	30.7	9.2
As 1 - 3 Post ht. treated	6 7 8	29.9 31.3 31.0	26.8 21.4 25.6	9.0 8.0 8.0	30.7	24.6	8.3
As 4 and 5 Post heat treated	9 10 11	33.3 33.1 33.1	29.6 29.6 30.7	10.0 10.0 10.0	33.2	30.0	10.0

MATERIAL DTD.546 ORIGINALLY 8 S.W.G.
ALL SPECIMENS NOMINALLY .080" thick.
ETCHANT 15% NaOH at 80°C.
POST HEAT TREATMENT 2 hrs. at 175°C.
.1% PROOF STRESSES ARE CHORD VALUES

* Spec. No. 3 Slipped in Grips ∴ .1% P.S. is ignored

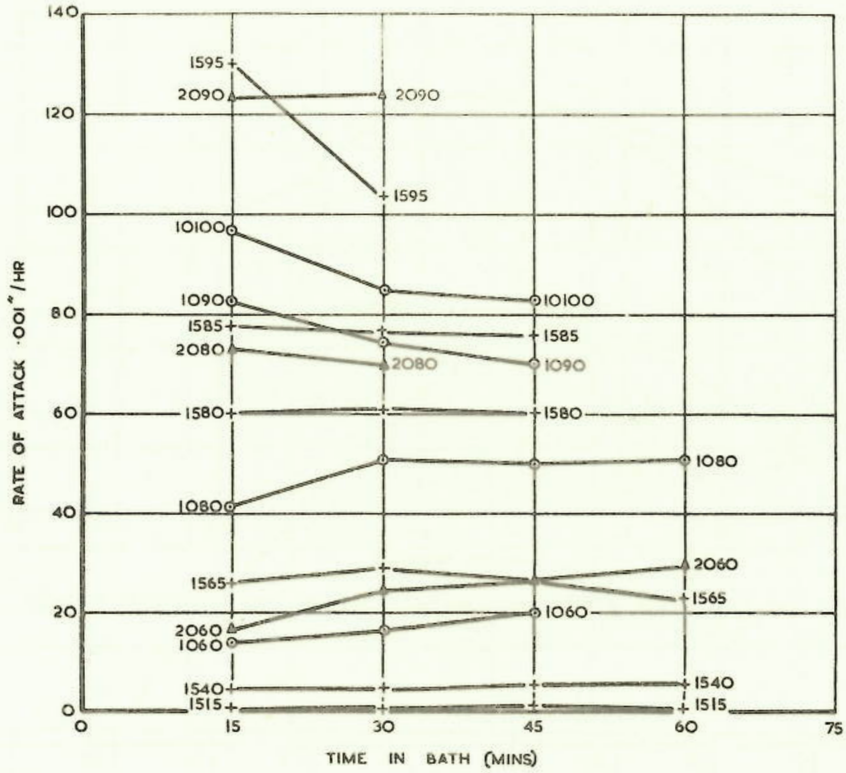

FIG. 1. VARIATIONS IN RATE OF ATTACK WITH TIME IN VARIOUS ETCHANTS

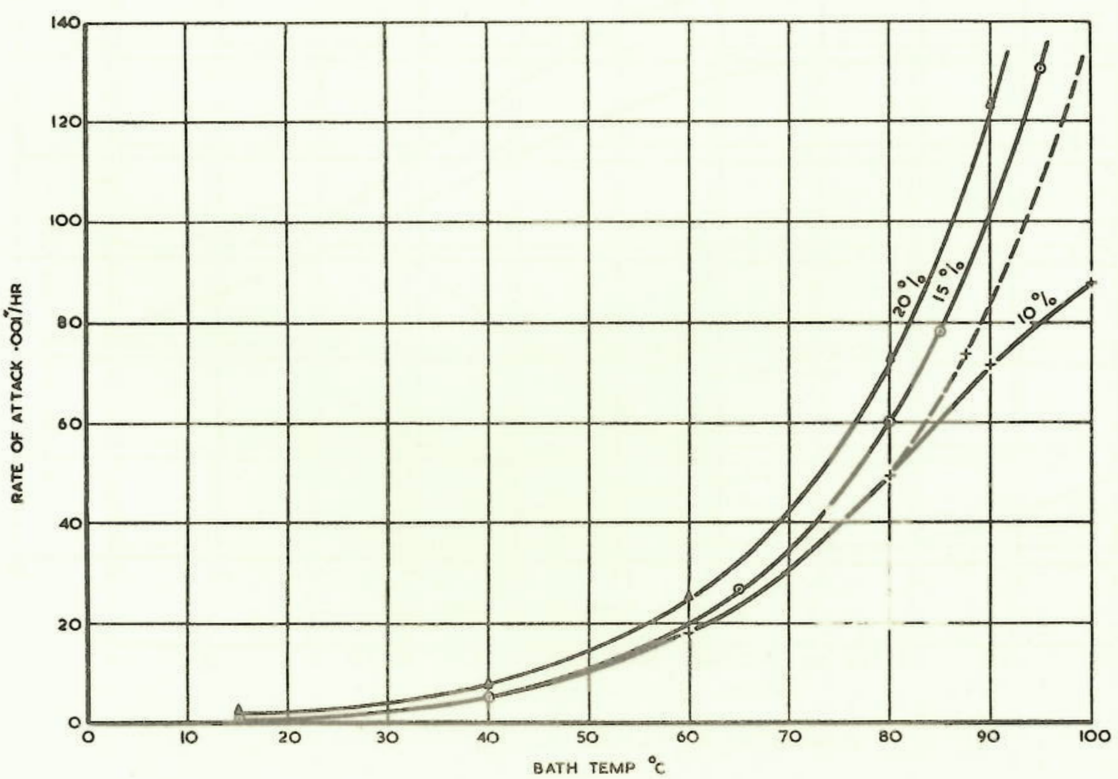

FIG. 2. EFFECT OF BATH TEMPERATURE ON RATE OF ATTACK IN VARIOUS CONCENTRATIONS OF SODIUM HYDRATE

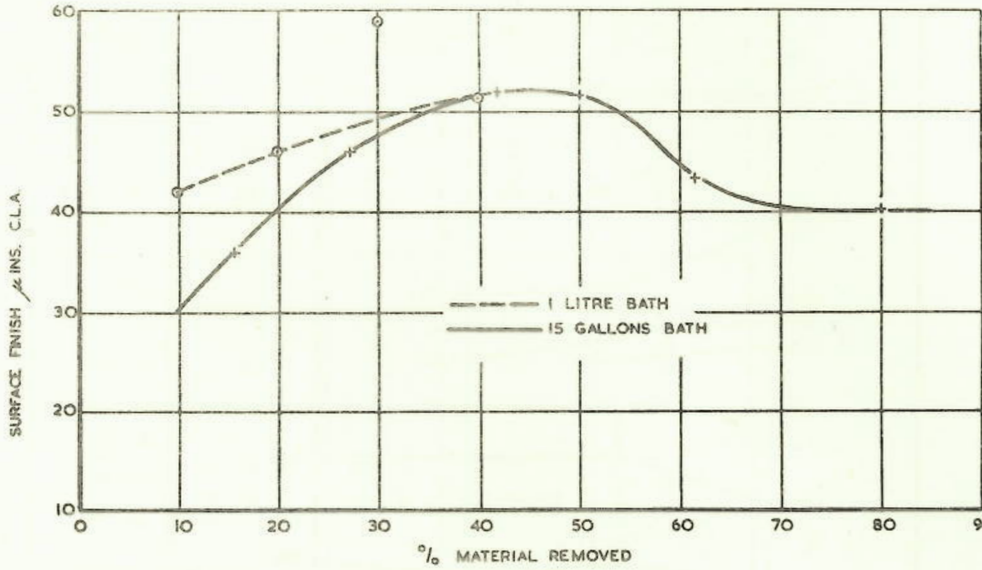

FIG. 3. EFFECT OF DEPTH OF ETCH AND BATH VOLUME ON SURFACE FINISH

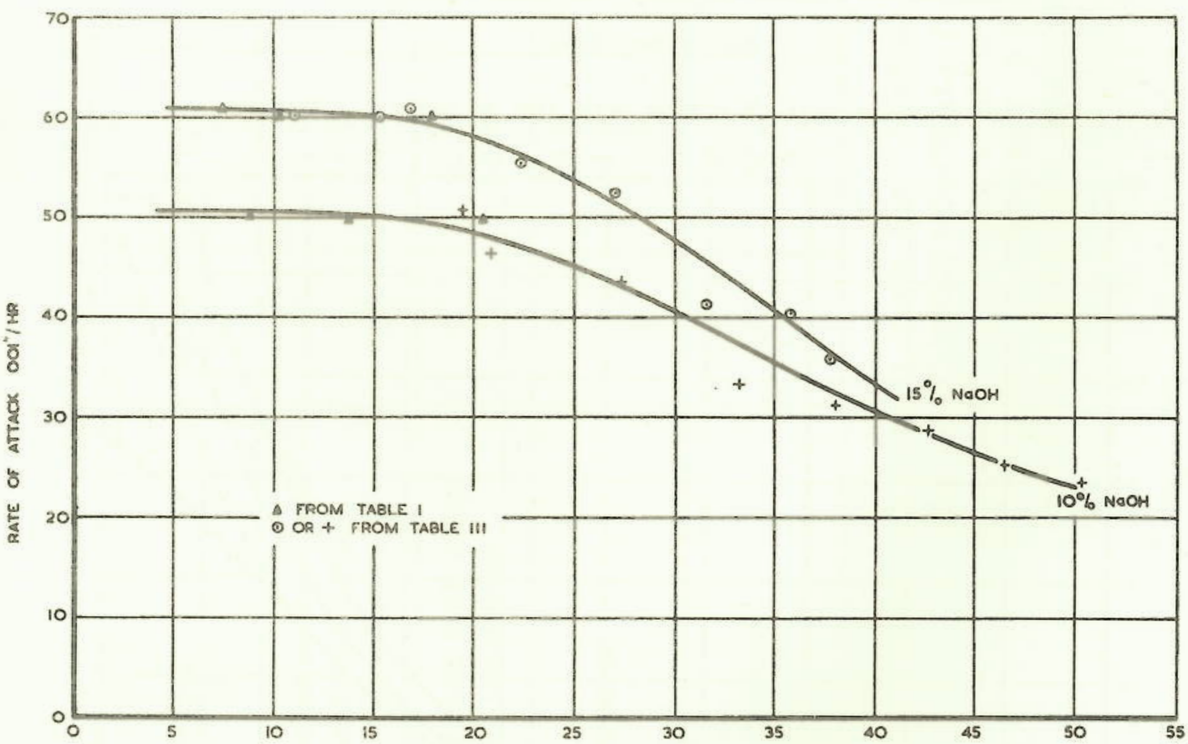

FIG. 4. ETCHANT BATH EXHAUSTION RATE

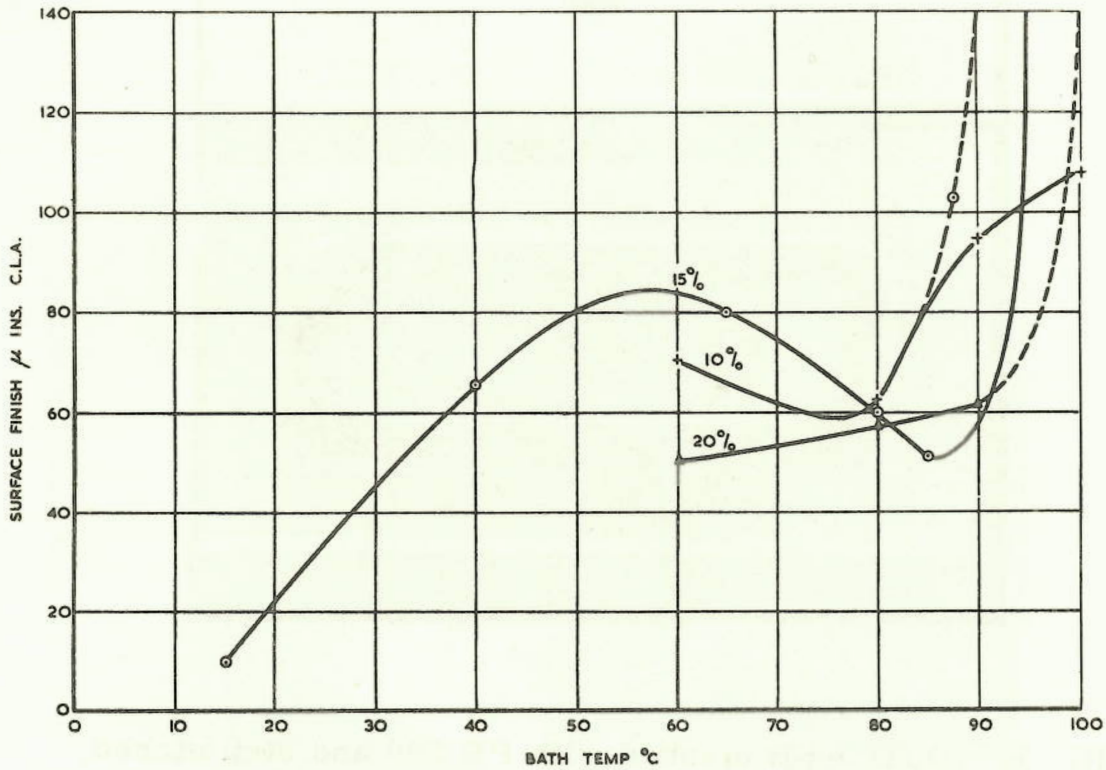

FIG.5. EFFECT OF BATH TEMPERATURE ON SURFACE FINISH IN VARIOUS CONCENTRATIONS OF NaOH

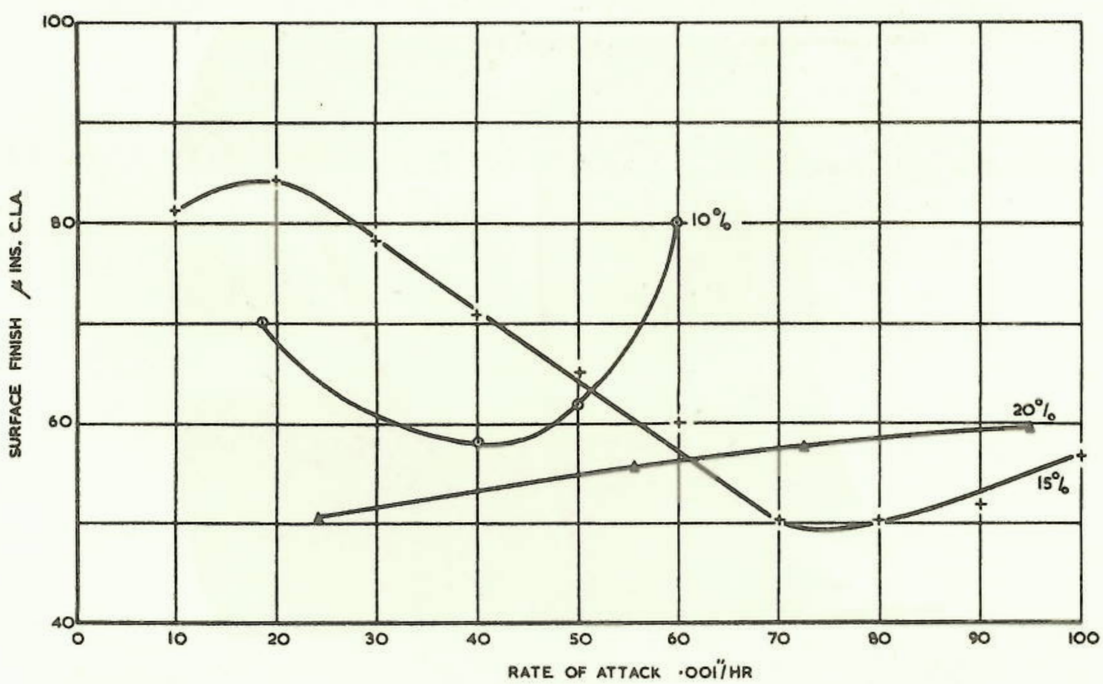

FIG. 6. REPLOT OF FIG. 2. AND 3.

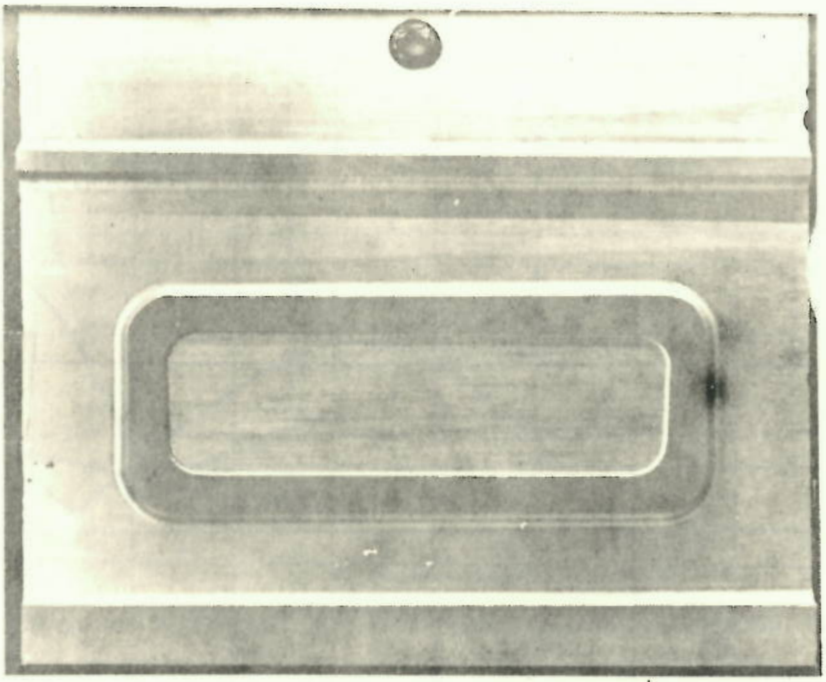

FIG. 7. DTD 546B masked with PR 500 and step etched.

FIG. 8. DTD 610B masked with PR 500 and formed after masking

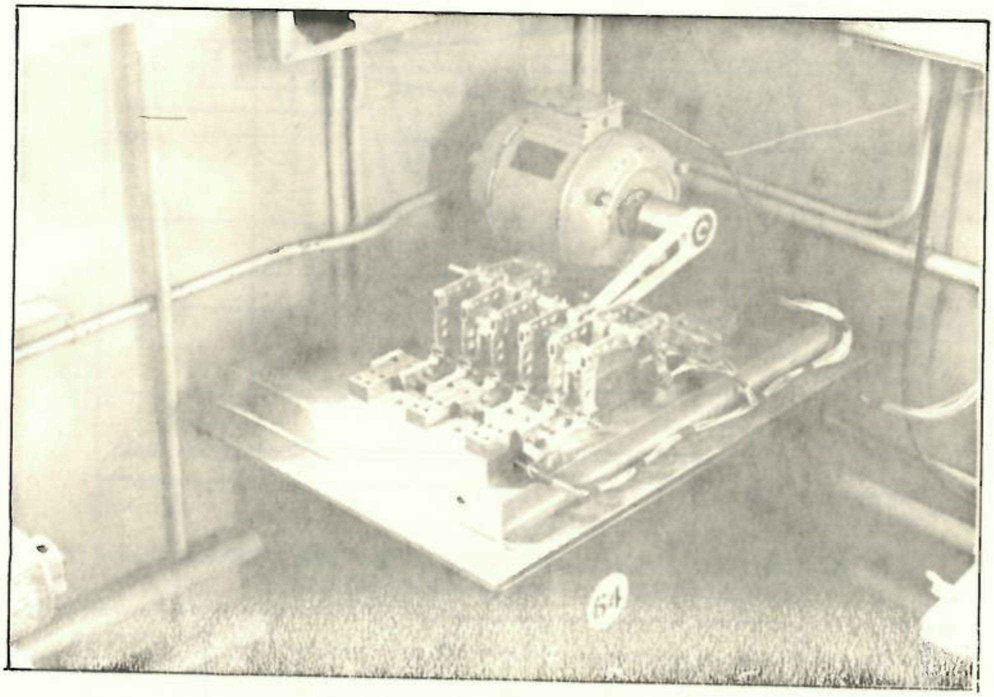

FIG. 9. Ten specimen reverse bend fatigue testing machine.

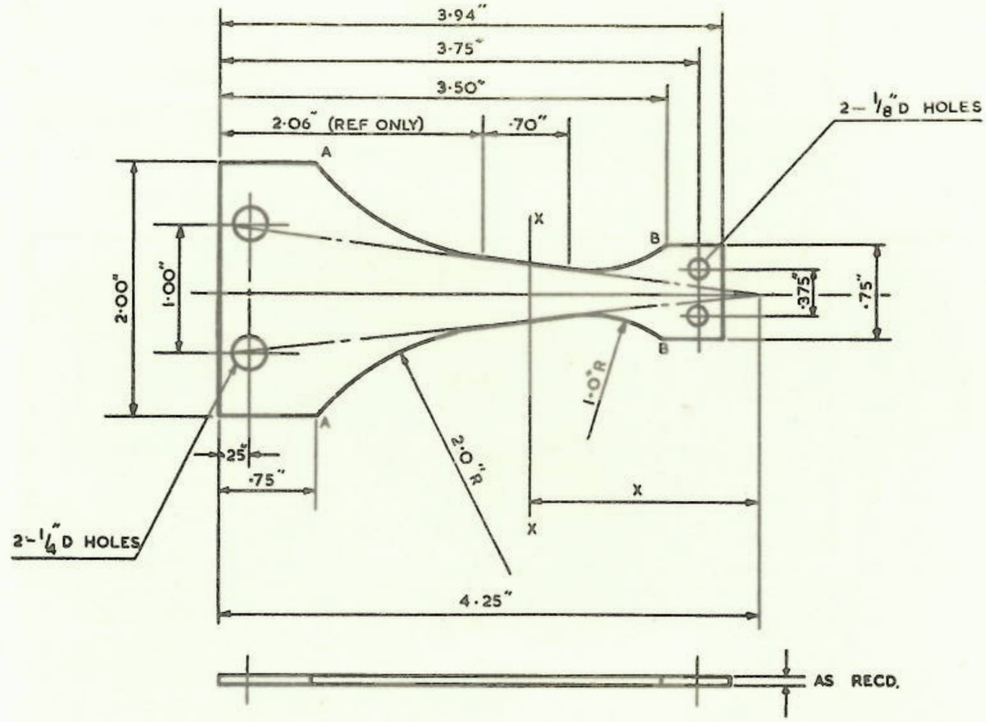

FIG. 10. FATIGUE TEST SPECIMEN

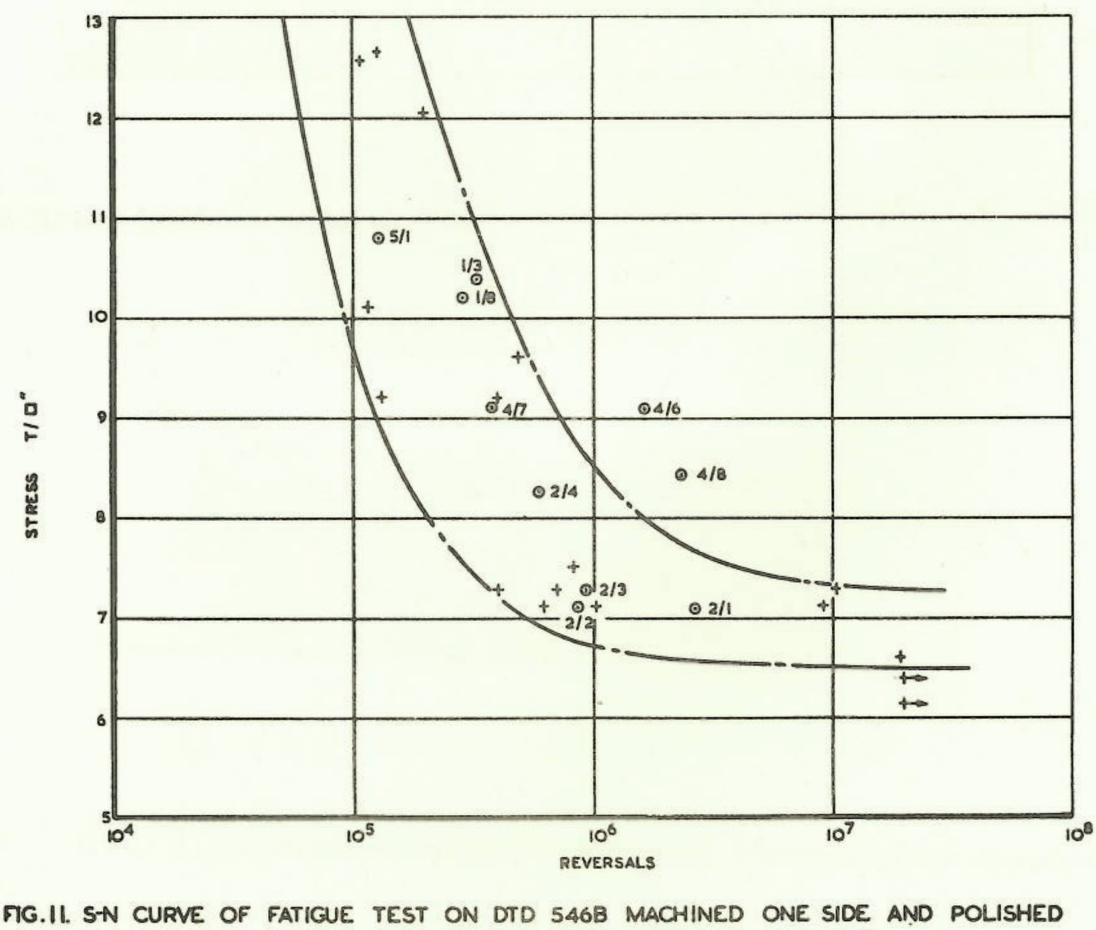

FIG. 11. S-N CURVE OF FATIGUE TEST ON DTD 546B MACHINED ONE SIDE AND POLISHED (TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

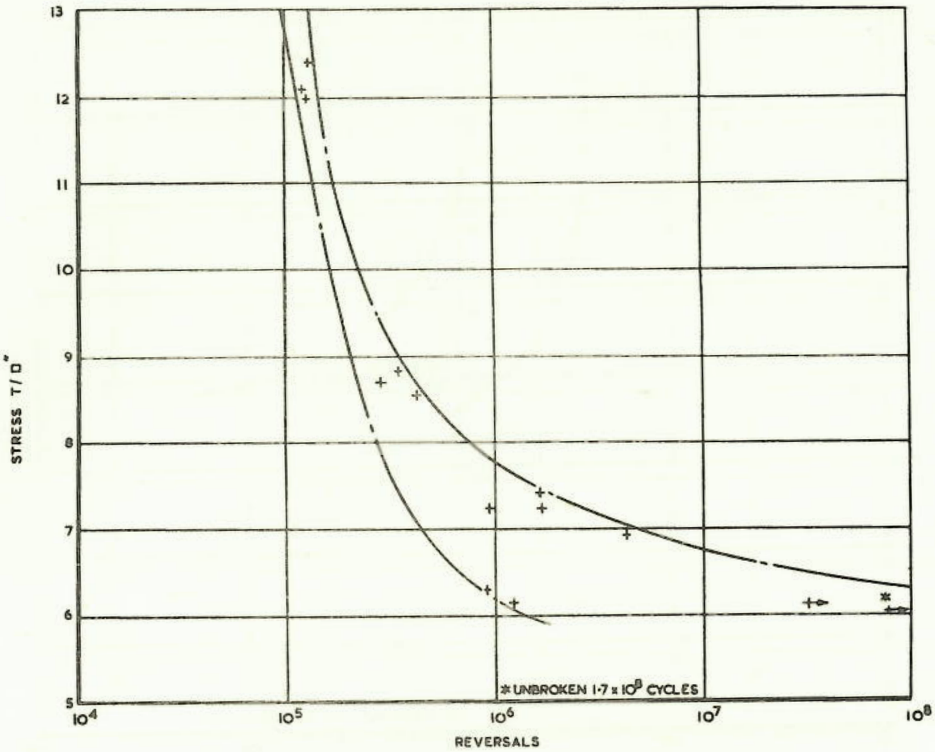

FIG. 12. S-N CURVE OF FATIGUE TEST ON DTD 546B FLY-MILLED ONE SIDE ONLY
(TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

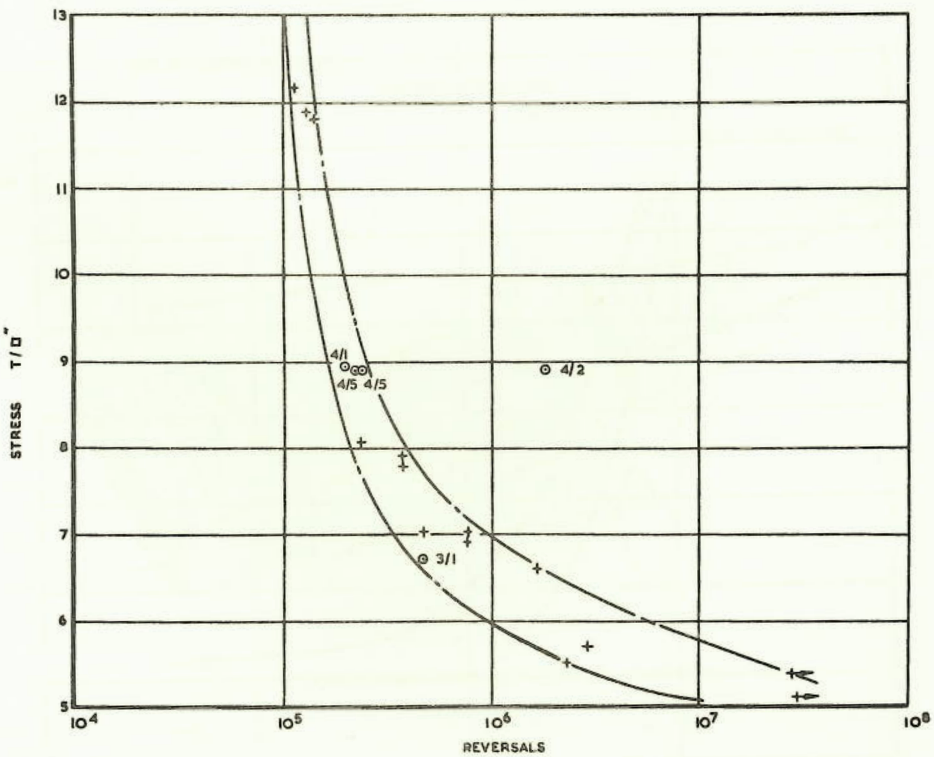

FIG. 13. S-N CURVE OF FATIGUE TEST ON DTD 546B CONTOUR ETCHED ONE SIDE ONLY
(TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

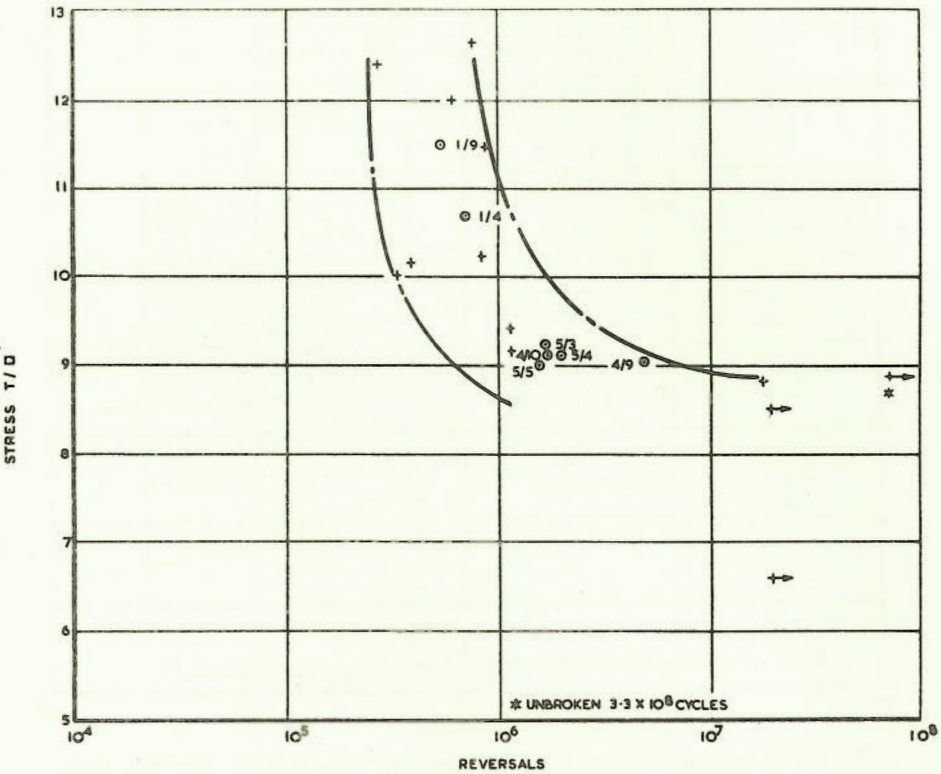

FIG.14. S-N CURVE OF FATIGUE TEST ON DTD 546B CONTOUR ETCHED BOTH SIDES (TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

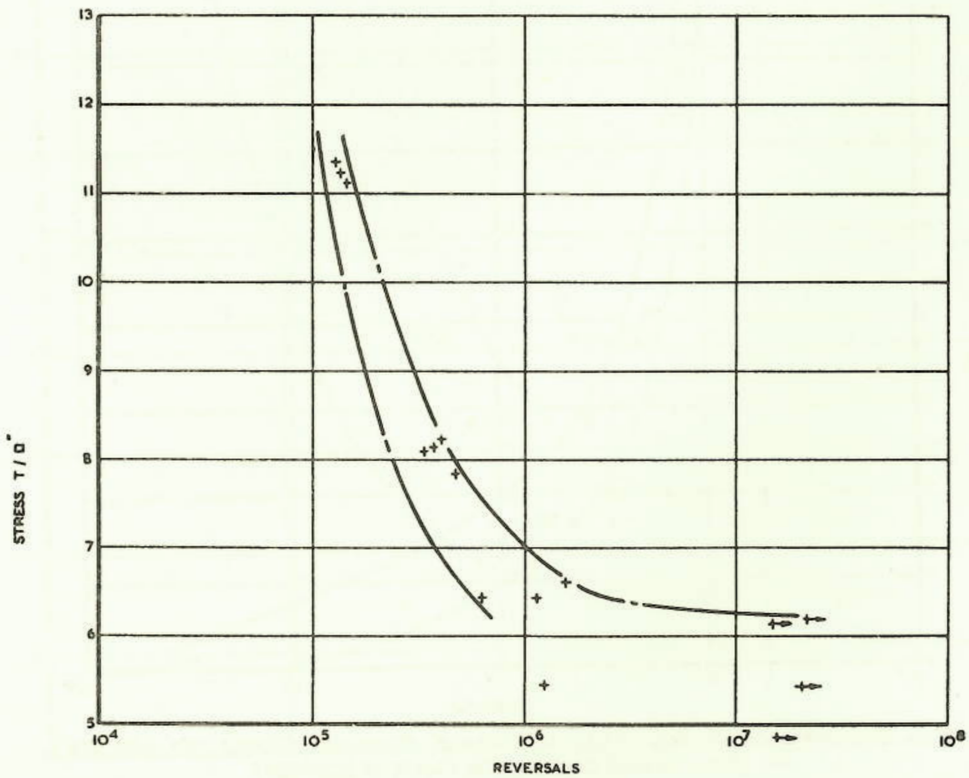

FIG.15. S-N CURVE OF FATIGUE TEST ON DTD 546B CONTOUR ETCHED ONE SIDE AND VACU-BLASTED (TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

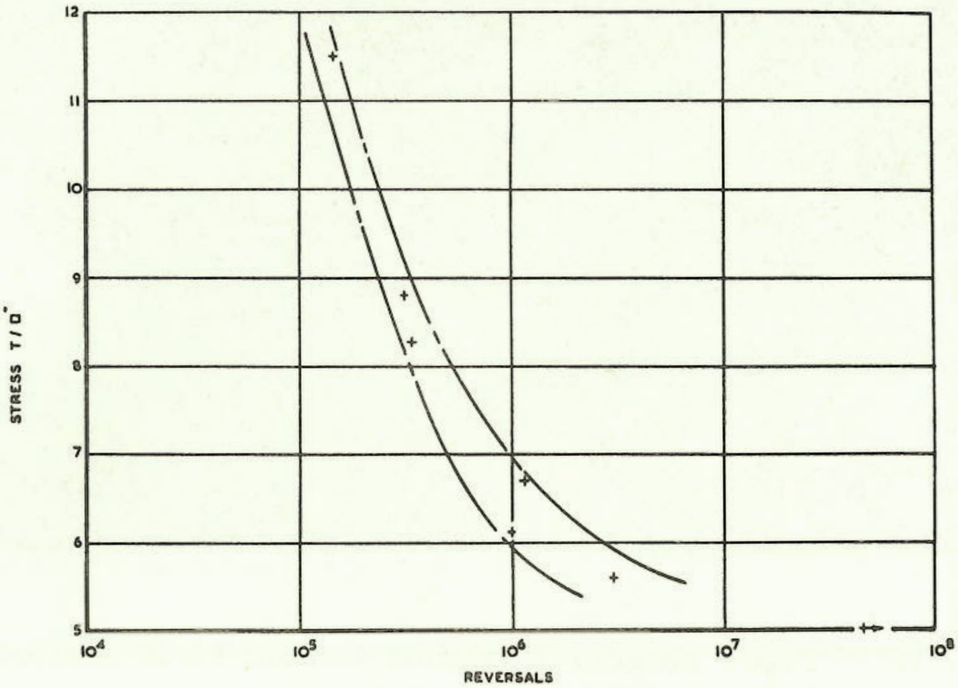

FIG. 16. S-N CURVE AFTER BOILING IN WATER FOR 30 MINUTES
(TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

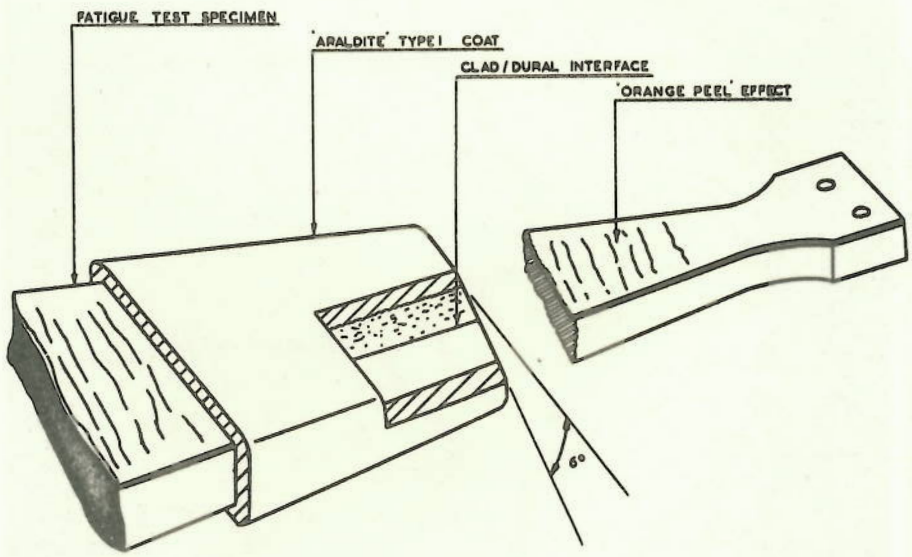

FIG. 17. POSITION OF TAPER SECTION

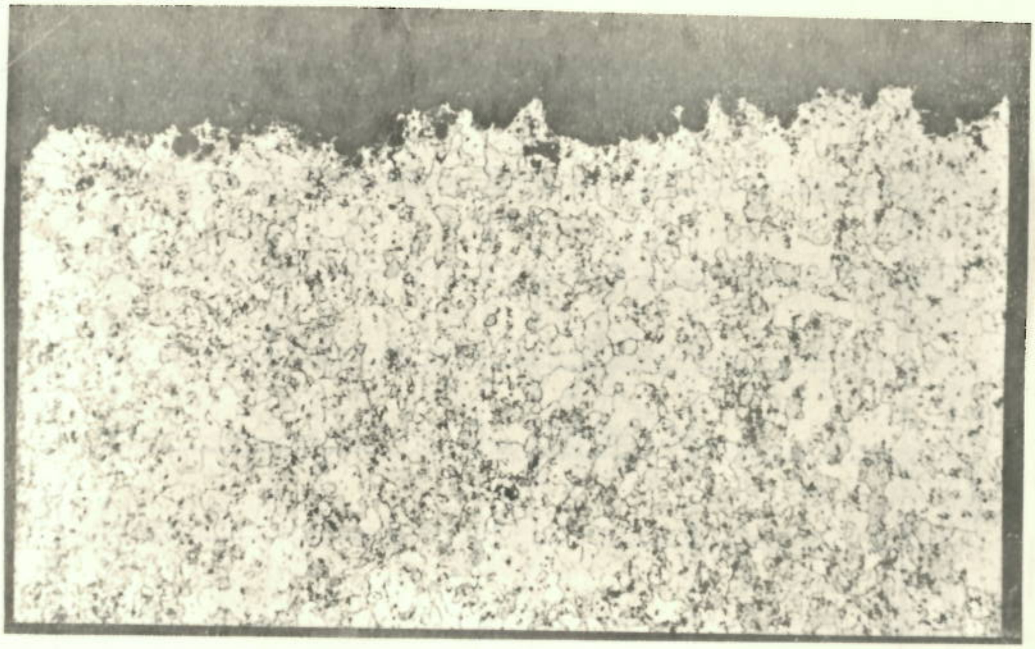

FIG. 18. Taper section of contour etched surface after fatigue failure. Micro-etched.

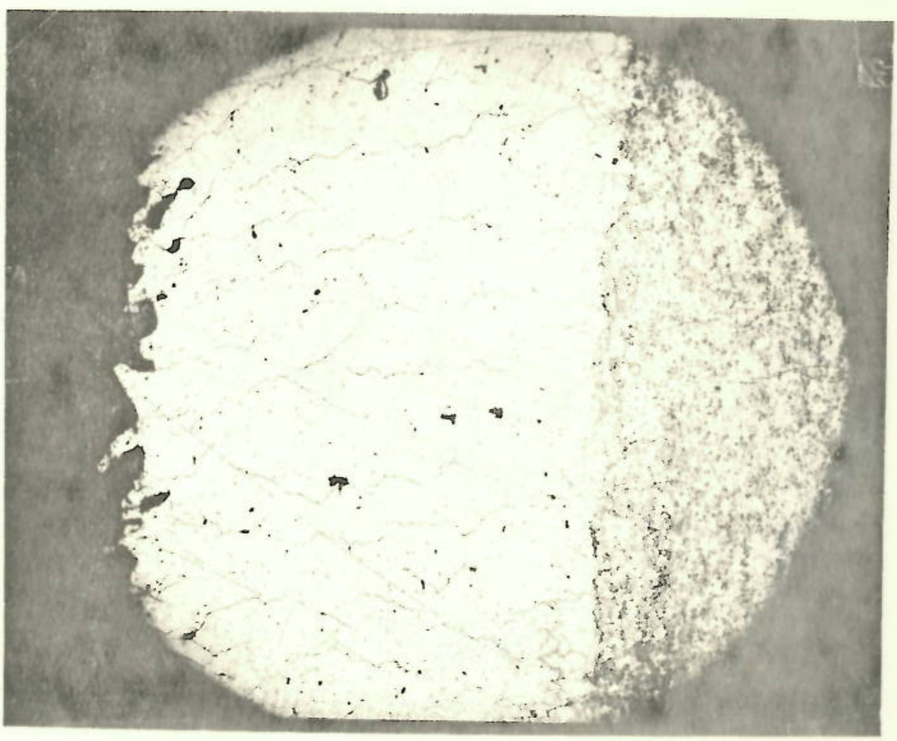

FIG. 19. Taper section of cladding and core of DTD 546B after fatigue failure. Micro-etched.

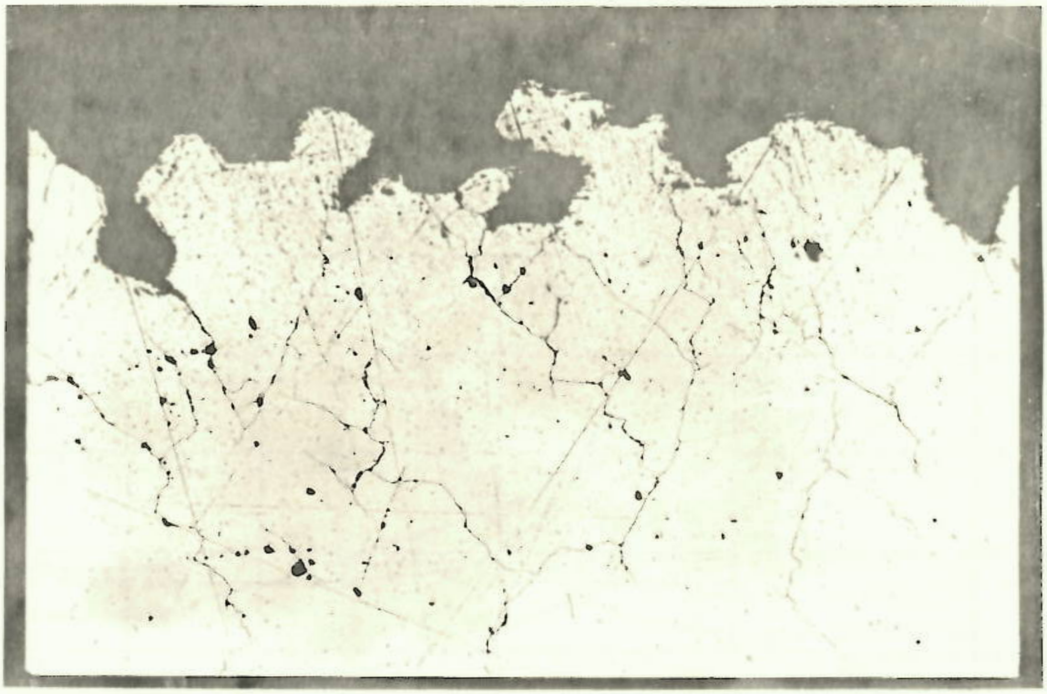

FIG. 20. Taper section of clad surface after fatigue failure. Micro-etched.

FIG. 22. Fatigue cracks in cladding.

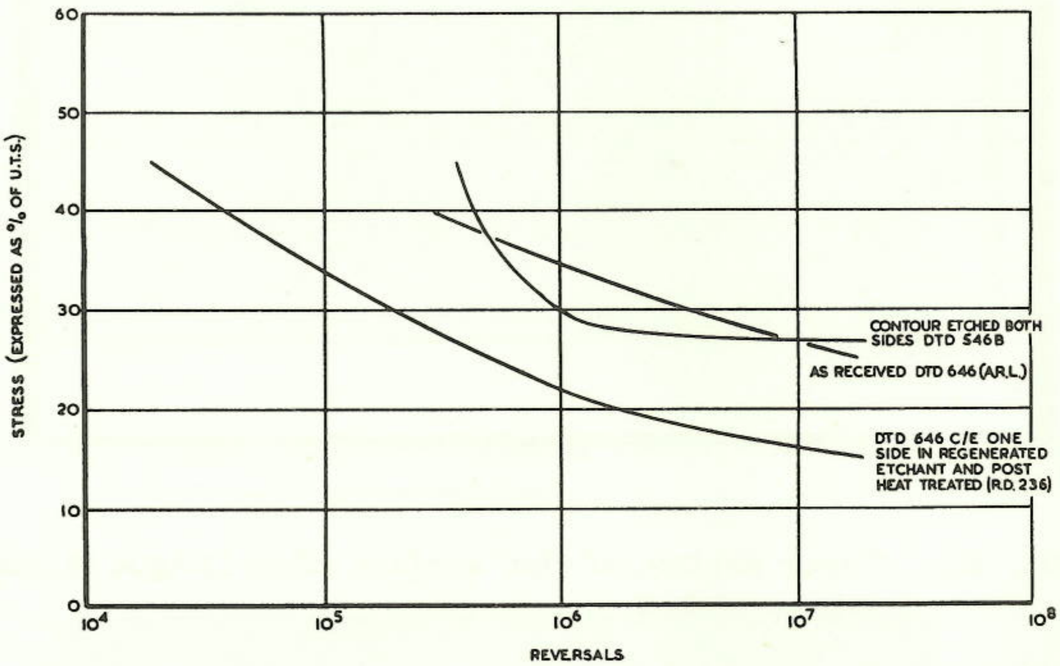

FIG.21. S-N CURVES FOR FATIGUE TESTS ON DTD 646 AND ON EFFECT OF CONTOUR ETCHING

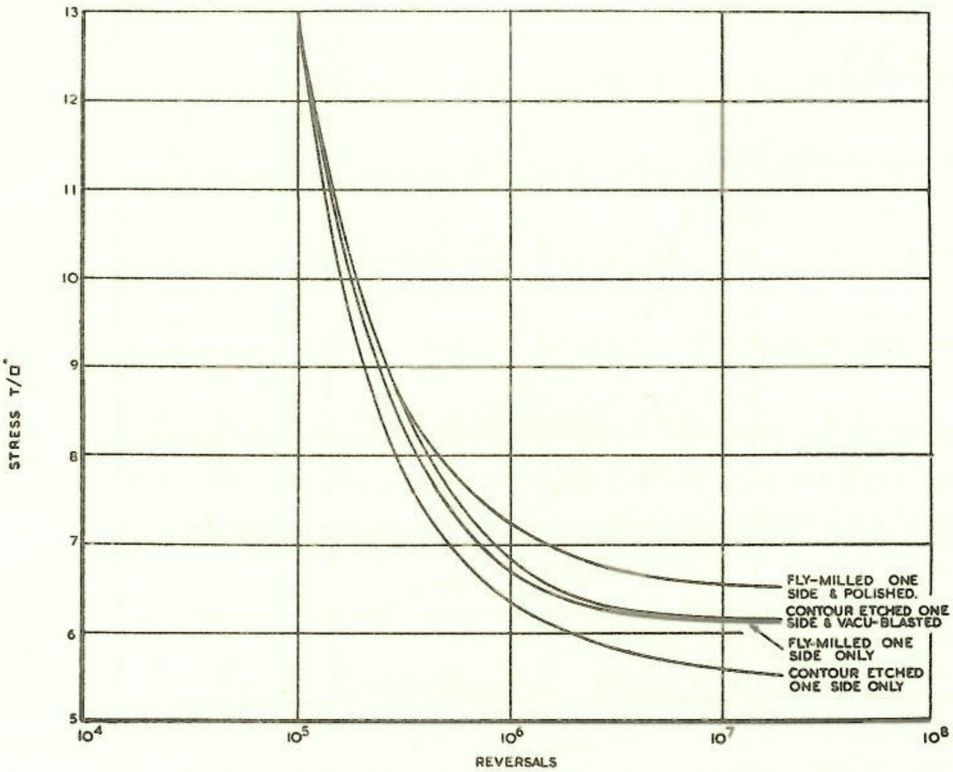

FIG. 23. COMPARISON OF S-N CURVES FROM FATIGUE TESTS ON DTD 546B AFTER VARIOUS SURFACE TREATMENTS

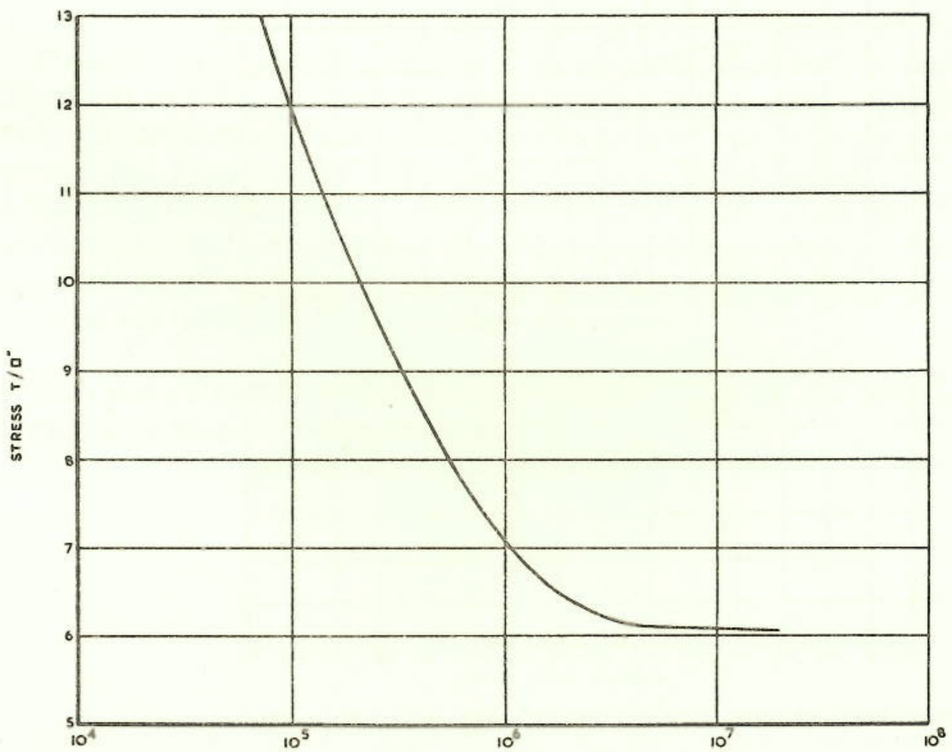

FIG. 24. S-N CURVE FOR FATIGE TEST OF DTD 546

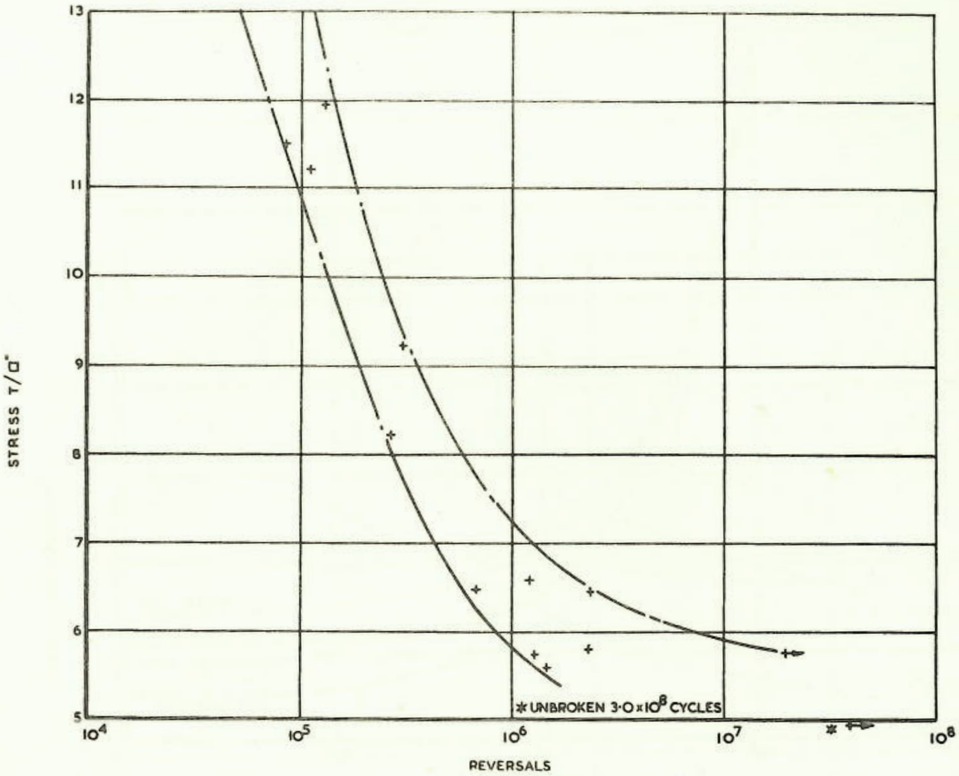

FIG. 25. S-N CURVE OF FATIGUE TEST ON DTD 546B MACHINED ONE SIDE AND POLISHED POST HEAT TREATED AT 175°C. (TWO CURVES DRAWN SHOW LIMITS OF SCATTER)

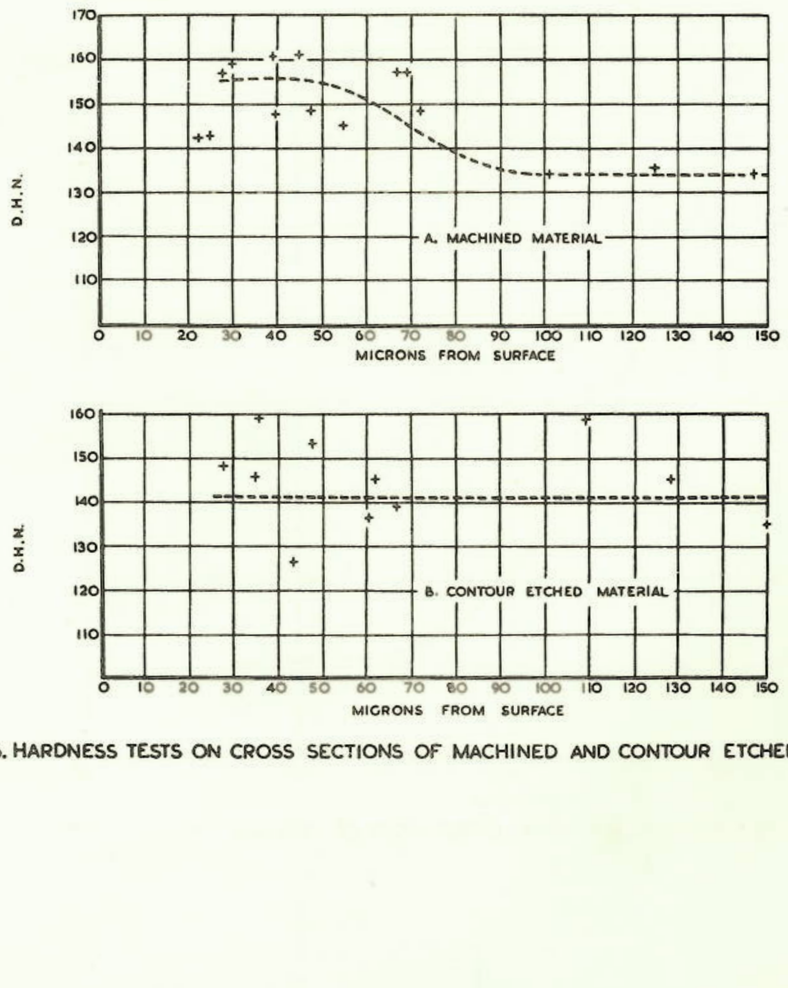

FIG. 26. HARDNESS TESTS ON CROSS SECTIONS OF MACHINED AND CONTOUR ETCHED FACES