

AERO RESEARCH, LTD.: TWENTY-FIRST ANNIVERSARY

THE firm of Aero Research, Ltd., at Duxford, Cambridge, owes its inception in 1934 to the urge of a university man—Dr. N. A. de Bruyne, at that time a Fellow of Trinity College, Cambridge—to carry out his own applied research work. This type of firm is probably commoner abroad than in Britain, although the Cambridge Instrument Co., Ltd., may be cited as another example.

During the first few years of its life as a small research unit, it pioneered a number of developments that have since proved their worth. These include structural materials for aircraft, honeycomb structures (British Patent 577,790 of 1938), the analysis of sandwich stabilization (*J. Roy. Aero. Soc.*, 44, 1; 1940) and the conception and realization in 1941 of the technique of metal-to-metal glueing for aircraft structures. This work on metal-to-metal adhesives called for both an investigation of the problems of adhesion (see, for example, *Aircraft Engineering*, 16, 115, 140; 1944. *J. Sci. Instr.*, 24, 29; 1947. *Research*, 6, 362; 1953) as well as the production problems presented by its almost immediate application in the de Havilland Hornet fighter. The techniques developed at that time remain the basis of the widespread use of 'Redux' glue in modern civil aircraft. The report on the Comet aircraft accidents unequivocally exonerated this method of construction and, in view of the conclusions reached by the inquiry, its use is to be welcomed as a method of reducing stress concentrations (Duncan, W. J., *Engineering*, 179, 196; 1950).

In all its projects the company has consistently emphasized the need for the research man to follow through to the final application of his work, as

evidenced even in 1934 by the building of a complete aeroplane to demonstrate the value of concepts, such as Wagner's tension field analysis, that were new at that time (*R. and M.* 1694).

On the outbreak of the Second World War the company made for the Ministry of Aircraft Production thirty Miles Magister tail-planes, and a Spitfire fuselage of plastic material; their performance on test (including flight trials of a tail-plane) was entirely satisfactory, but as the shortage of light alloys was overcome, the need for substitutes disappeared. With the rise in aircraft speeds that has since taken place, however, interest in plastics for aircraft has considerably revived. A war-time development that considerably accelerated the production of wooden aircraft and gliders was the 'strip heating' process for which the Royal Commission on Awards to Inventors made an *ex gratia* award.

Until 1946 the company had been wholly owned by Dr. N. A. de Bruyne, but in that year a majority shareholding was taken up by Ciba, Ltd., the worldwide organization known for its research in dyestuffs, pharmaceuticals and plastics. Under an arrangement with Ciba, Ltd., Aero Research, Ltd., continued under its own board as an active research centre, as well as becoming a large production unit for 'Araldite' epoxy resins and the 'Aerolite' urea formaldehyde adhesives which the company had been making since 1937.

The recent visit of H.R.H. the Duke of Edinburgh on April 20 provided a fitting occasion to celebrate the twenty-first birthday of the company by a display of research and production, including some of the new high-temperature adhesives which are now under development.

FATIGUE IN METALS

AT a joint meeting in the Clarendon Laboratory, Oxford, on April 15, of the Oxford Local Section and the Metal Physics Committee of the Institute of Metals, the morning session was devoted to a discussion on "Fatigue". Prof. G. V. Raynor (Birmingham) presided, and invited papers were given by Dr. N. Thompson (Bristol) and Dr. J. Holden (National Physical Laboratory, Teddington). Dr. Thompson first mentioned the usual analysis of a fatigue experiment into three stages, consisting respectively of a period of rapid work-hardening, a long secondary stage in which local deformation may increase slowly but nothing much seems to happen, and a short third stage in which a crack is rapidly propagated, leading to failure. In contrast to this, recent work suggests that the formation and growth of the crack occupy a considerable part of the second stage, and Dr. Thompson devoted most of his introductory talk to a discussion of the evidence for what he described as this still slightly heterodox view.

In specimens containing notches, it is generally accepted that a crack may be present from an early stage in the fatigue life. The difficulty here is to explain why this crack does not spread rapidly, and no very convincing reason has yet been given. In plane specimens, without deliberately introduced

stress raisers, cracks are not normally visible until just before failure. A theoretical model, due to Head, suggests that the length l of a crack is given by a formula of the type

$$l^{3/2} = 1/A(N_{\infty} - N)$$

where N is the number of cycles, and N_{∞} the number of cycles to failure. The rate of growth of the crack is thus proportional to $l^{3/2}$, and since this varies so rapidly, it is possible that for a large part of its life the crack may be too small to be visible. The few experimental measurements which have been made are in agreement with Head's equation.

Dr. Thompson next mentioned some experiments by Sinclair and Dolan, in which fatigue tests on α -brass were interrupted every 20 per cent of the estimated life in order to give the specimen a half-hour annealing treatment at 400° C. These anneals had no effect on the fatigue life, although the specimen was completely resoftened at every stage. Sinclair and Dolan suggested that the only type of defect which would survive the annealing treatment was a small crack, which must thus have been formed in the first 20 per cent of the life. Similar experiments at Bristol in which copper specimens were annealed

at 600° C. every 10 per cent of the estimated life produced the same result.

In recent work at Bristol, specimens of high-purity copper were given a careful surface preparation and finally electropolished before testing in push-pull at 1,000 c./s. The specimens were removed and examined at various stages in the experiment. It was found that the eventual fatigue crack always originated from an intense slip band formed in the early part of the deformation; the path of the crack in both directions from this point was shown by a very high density of slip markings, while the central portion was relatively free from slip. To reduce difficulties caused by the complex nature of the surface markings, the specimens were re-electropolished at various stages during the experiments, a standard technique allowing the removal of $\sim 2\mu$ of material. This polish was found to remove nearly all the slip markings, but a few remained and are called 'persistent' slip bands. Persistent slip bands appeared after about 4 per cent of the fatigue life, and increased in number during the experiment; after about 40 per cent of the life, some of them had definitely spread into neighbouring grains, and after 70–80 per cent of the life they were recognizable as small cracks. Application of a large tensile stress to a specimen containing a number of persistent markings resulted in the opening out into visible cracks of all the markings which extended over more than one grain diameter. These markings thus represent micro-cracks, and the evidence strongly suggests that cracks form at a very early stage in the fatigue process. Dr. Thompson ended by pointing out there is some uncertainty in the description of a very small crack in the early stages of the process; the question of whether or not the persistent markings are micro-cracks when they are less than one grain diameter in length may be partially a matter of definition.

Dr. Holden's paper was concerned with the now popular view that recovery processes occur during the course of fatigue, even at room temperature. Specimens which are normally metallurgically stable thus become unstable during fatigue conditions, and diffusion and related phenomena may be greatly increased. The effect on precipitation was illustrated by recent experiments at the National Physical Laboratory on iron, copper and commercial aluminium, in which a progressive loading technique was used. The behaviour of the iron and aluminium specimens was essentially similar, and indicated that greatly enhanced ageing occurred, especially in the aluminium.

A more direct indication that diffusion is facilitated during fatigue is provided by experiments on the internal oxidation of copper alloys containing 0.01 per cent silicon. The process of internal oxidation depends on the inward diffusion of oxygen atoms through a surface sub-scale layer, and the diffusion of silicon atoms within the alloy. Under static conditions, it is known that periods of about one month are required to produce a detectable sub-scale at 300° C. Fatigue tests at this temperature resulted in oxide precipitation within the gauge length after 200–300 hr., although there was no sign of precipitation within the unstressed parts of the specimens.

Having demonstrated the existence of enhanced diffusion effects, Dr. Holden finally outlined experiments now being undertaken with the object of determining the type of lattice defect which is responsible. Low-angle X-ray diffraction studies are being made on 50- μ copper foil specimens given

pulsating tension fatigue tests. After testing for less than 5 per cent of the fatigue life, very pronounced low-angle streaks are found, and these appear to be generally similar to the streaks observed in the very early stages of ageing. The streaks vary from grain to grain, and are unaffected by annealing the foil for an hour at 150° C.

In the general discussion, some attention was paid to the nature of the persistent slip markings. Dr. Thompson emphasized that they are retained after annealing, and are not simply large steps, since the standard electropolishing treatment has been found to remove the slip bands formed by static tensile deformation. Mr. M. Mallon (Shrivenham) said that persistent slip markings were formed when α -brass was deformed more than 20 per cent, but closer examination showed them to be composed of very small etch pits. Mr. G. Smith (Cambridge) described work on fatigue tests at stresses below the fatigue limit. Etch-pits were found to remain in regions where large slip bands had been visible before polishing; on testing at higher stresses, these linked up together to form markings.

In reply to a question, Dr. Thompson said that the cracks in his specimens always originated from the free surface, even though push-pull testing was used. He described further experiments in which 20–30 μ of surface material was removed, and the persistent slip markings then disappeared. The markings on one specimen were removed in this way after ~ 25 per cent of the estimated life, and testing at the same stress was then resumed. This procedure was repeated seven times in all, so that the specimen had lasted for 225 per cent of its normal life, and there seemed no reason to doubt that the process could be continued until there was no specimen left.

Dr. R. T. Parker (Banbury) pointed out that Dr. Thompson's results implied that the grain size should be an important factor in fatigue; there was general agreement on this, but critical data seem to be lacking. Dr. A. N. Turner (Banbury) raised the question of fatigue in non-metallic materials, and also asked about experiments on single crystals. Dr. Thompson said that experiments on single crystals at Bristol had given essentially similar results to those on polycrystalline material; the crack again originated in an intense slip band. Dr. E. G. Stanford (Banbury) mentioned Hanstock's work on fatigue, in which internal friction measurements were used. The results did not seem to agree with Dr. Thompson's work. Dr. Thompson pointed out that these experiments involved non-uniform stresses, and were made on precipitation-hardening alloys, which are metallurgically unstable under fatigue conditions. Experiments at Bristol have shown that the internal friction is associated with the total number of visible slip bands; it is markedly affected by annealing, but not by the surface dissolution. There is thus no direct correlation between internal friction and fatigue.

In reply to a question, Dr. Holden amplified his remarks on the low-angle X-ray experiments. By analysing the distribution of X-ray intensity, it is hoped to distinguish between effects caused by arrays of dislocations and those due to excess concentrations of lattice vacancies. The session concluded with a description by Dr. H. M. Rosenberg (Oxford) of recent work on fatigue at the Clarendon Laboratory. *S-N* curves for brass and copper have been determined at room temperature, 90° K. and 20° K. At a given stress level, the fatigue lives were in the approximate

ratios 1:10:100. More significantly, the stress required for failure after a given number of cycles increased in the same ratios ($\sim 1:1.5:2$) as the static ultimate tensile strength. Work at liquid helium temperature, now proceeding, shows still further increase in fatigue life, although the correlation with the change in ultimate tensile strength is no longer valid. No one seemed to have a ready explanation for these rather surprising results.

The afternoon session of the meeting was in the form of a general discussion, in which a panel of members of the Metal Physics Committee attempted to answer questions suggested by the local section. The current interest in fatigue problems was shown by the question, "Why have very high-strength aluminium alloys a fatigue strength no better than that of medium-strength alloys?", which produced, in effect, a continuation of the morning discussion.

J. W. CHRISTIAN

AROMATIC SUBSTITUTION

ON March 29 a symposium was held by the Chemistry Department of Queen Mary College, London, on "Aromatic Substitution", with Prof. E. R. H. Jones in the chair. The six contributions covered various aspects of electrophilic, nucleophilic and homolytic substitution.

Prof. M. J. S. Dewar reviewed the general theory of aromatic substitution in terms of the Wheland transition-state treatment and the molecular orbital method. The latter method may be used to calculate the position of substitution in polycyclic alternant hydrocarbons, and the results obtained are generally in agreement with the experimental results. The theory does not take into account steric hindrance, which appears to occur particularly in α -naphthalenic positions such as the 4-position in phenanthrene. Electrophilic substitution in heterocyclic systems was then discussed and the molecular orbital theory shown to be applicable in this field. Finally, Prof. Dewar described work being carried out on the nitration of quinoline which shows that this reaction proceeds by different mechanisms in acetic anhydride and sulphuric acid solution. In the subsequent discussion Prof. E. D. Hughes emphasized the importance of the reagent and also the effect of temperature on the orientation of the substituent. Prof. Dewar agreed and illustrated the effect of the reactivity of the reagent.

Dr. G. Baddeley¹ discussed the Friedel-Crafts acylation and alkylation of aromatic compounds, and emphasized the effect of the reactivity of the reagent on the nature of the product. With aluminium chloride in methylene chloride solution the usual orientation rules were observed, whereas if the solvent was nitromethane or ether the reagent was less reactive and 'abnormal' products were obtained. This was illustrated with numerous examples such as the substitution of anthracene, which occurs in the 9-position in methylene chloride solution but in the 1- and 2-positions in nitromethane solution. Dr. Baddeley ascribed the abnormal orientation to complex formation between the catalyst and solvent, attack by the bulky complex being liable to steric hindrance. Dr. E. A. Braude suggested that reversibility of the reaction might account for the abnormal orientation in polar solvents, but Dr. Baddeley said that, if excess aluminium chloride is avoided and the

hydrogen chloride removed, the reactions are not reversible.

Dr. P. B. D. de la Mare then read a paper on the chlorination and bromination of aromatic compounds. He stated that these reactions, when carried out by a solution of the halogen in hydroxylic solvents, can be shown kinetically to involve molecular halogen, but that brominations by aqueous hypobromous acid have a kinetic form which implies that the electrophilic reagent carries a full positive charge. Dr. de la Mare then compared molecular halogenation with ionic nitration and showed that the results suggest that, while the positively charged reagent evokes the inductive effect of the substituent most powerfully, the molecular reagent evokes the conjugative electromeric effect more powerfully.

The afternoon session was opened by Dr. N. B. Chapman, who gave a short survey of nucleophilic substitution and then proceeded to discuss the nucleophilic displacement of halogen. He directed attention to possible duality of mechanism (analogy with saturated systems) and to the possibility of complexity of mechanism in bimolecular processes, and outlined the structural requirements for nucleophilic substitution in homocyclic aromatic compounds. Dr. Chapman then discussed the quantum-mechanical background to the nucleophilic displacement of halogen in heterocyclic compounds (mono- and di-aza-naphthalenes) and gave an account of some kinetic investigations which have provided kinetic results for comparison with the approximate quantum-mechanical calculations; the agreement between the calculated and observed activation energies is not very good. In the discussion Dr. E. Parker suggested that the structure of the transition-state might involve *d*-orbitals, and this suggestion was generally accepted as being probable. Prof. Dewar suggested that a comparison of calculated activation energies with observed rates of reaction might be more correct and lead to a better agreement between theory and experiment.

Prof. W. Bradley discussed nucleophilic substitutions in which hydrogen is replaced by an anion, the formation of *o*-nitrophenol from nitrobenzene and potassium hydroxide being a typical example. The reactivity of the nucleus towards nucleophilic substitution decreases from anthracene to naphthalene to benzene, and nucleophilic substitution is facilitated by a carbonyl or nitro group attached to the nucleus, the positions *ortho-para* to the group being preferentially attached. Numerous examples show that, on direct hydroxylation with potassium hydroxide, mono-ketones and di-ketones yield respectively mono- and di-hydroxy compounds, and substitution by other anions follows a similar course. Prof. Bradley then gave several examples where substitution by an anion is accompanied by self-union of two molecules of the aromatic reactant, usually via a carbon atom *para* to a carbonyl group. He expressed the view that this is due to the anion functioning as a base and removing a proton from the aromatic reactant.

Prof. D. H. Hey opened his paper on homolytic substitution² by outlining the reactions by which phenyl radicals may be generated, and he then briefly discussed the partial rate factors for the phenylation of a series of mono-substituted benzene derivatives. He described the phenylation of toluene, ethylbenzene, isopropylbenzene and *tert*-butylbenzene; with the first three of these compounds the reactions are complicated by dimerization due to side-chain attack.