Rintech strengthens its contacts with industry...

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Mintech strengthens its contacts with industry . . .

Explosives Establishment open day reveals an 'arsenal' of polymer and allied developments

THE Ministry of Technology opened the doors of its Explosives Research and Development Establishment at Waltham Abbey last month, literally with a bang, and revealed that as well as explosives and rocket propellants it also has a minor arsenal of polymer developments to talk about.

Stemming in many cases from military research, but now reaching a point of more general industrial interest, these developments include advanced work on fibre reinforcements for plastics, a new approach to synthesising tailor-made polymers, a new conductive natural rubber compound and some interesting ideas on interchangeable mould cavities and steel-filled epoxy injection moulding tools.

Work on polymers now constitutes some 30% of the total effort at Waltham Abbey, and the most important area is that of reinforcement, where ERDE has been investigating asbestos, glass, carbon fibre and "silicon-based 'whiskers'.

Effort has been concentrated on processing these fibres to render them more suitable for reinforcement of plastics, and ERDE has now come up with a process for grading or aligning fibres to give better distribution, orientation or 'packing' in plastics composites.

The development programme has shown that the length/diameter ratio of the fibre is important in determining the degree of reinforcement obtainable. The longest fibres are used in thermosetting resins and the shorter ones in thermoplastics and light alloys.

On grounds of cost and potential general usefulness, ERDE has singled out asbestos for special attention as a reinforcement and polypropylene as a host matrix.

This is not in itself new, since there are commercial grades of asbestos reinforced pp in the USA, where they have recently been enjoying some popularity for automobile components, and it is understood that a UK producer will be introducing an asbestos-filled grade officially later on this year.

Moreover, at the recent Plastics Institute conference on reinforced plastics (PRW May 17), G. L. Wicker, of Turner Bros, saw the future lying strongly with thermoplastics, with asbestos as a major reinforcing fibre.

But, in practice so far, a major problem has been to obtain an even distribution of the fibre in the thermoplastic matrix — and it is this problem which ERDE now claims it has solved with its grading process.

Another factor is, naturally, the ability of the resin to wet the fibre (which influences the all-important fibre/resin bond), and ERDE is tackling this on two levels — pretreatment of the fibre and the addition of specific reactive groups to certain polymers to try to improve adhesion.

Results so far show that breaking strengths of thermoplastic mouldings can be increased from 100% to 200% and flexural modulus by up to 500% by the incorporation of about 30% by weight of asbestos.

In polypropylene, for example, flexural modulus is increased from 0.15 unfilled to 0.77 (psi x 10^6) by the addition of 40% asbestos, and tensile strength from 4,400psi to 6,800psi. With nylon 66 (where a direct comparison with commercially-available glass-filled grades is possible), flexural and tensile figures are quoted as 0.3 and 11,000 (unfilled), rising to 0.8 and 18-24,000 (glass-filled) and 1.3 and 19,000psi (30% asbestos-filled).

STIFFNESS

In comparison with glass fibre — the main fibrous reinforcement for thermoplastics at present — asbestos gives a higher stiffness but a lower tensile strength, but it is argued that, for a large majority of injection moulding applications, stiffness rather than tensile strength is the more desirable property.

Where this is the case it is possible that a thinner section could be employed, thereby economising on material, but the real argument put forward by ERDE (which has no particular axe to grind) is that graded asbestos, at an estimated 2s to 3s a lb, is cheaper than glass fibre at 3s to 4s a lb.

From experience so far, the ERDE team finds that most of the wide range of thermoplastics it has so far reinforced with asbestos can be processed satisfactorily on conventional screw injection moulding machines.

The exception is styrene-based materials (which can give moduli up to 6 x 10^6psi), which have to be moulded by ram injection or compression techniques. Wear and tear on the machine is said to be negligible after 18 months' trials.

For thermosetting resin reinforcement, ERDE has developed alignment processes, to orientate the fibres and pack the maximum amount into a composite.

DISPERSION

Both processes depend on dispersion of the fibre in a carrier (which may be a viscous aqueous solution of ammonium alignate in water, or may be glycerine), alignment and removal of the carrier. The basic technique for alignment is the use of viscous drag in extrusion through an orifice or slit.

These techniques can be used to produce aligned reinforcement in the form of composite sheet (for bars, tubes and shapes produced in heated presses for high performance structural applications), thin composite sheet (5-10thou thick for honeycomb structures where weight is important), and thin narrow continuous composite strip (for winding techniques for tanks, tubes and similar regular-shaped items).

Composites have so far been made from phenolics, polyesters and epoxies, applying the resin to the aligned reinforcement in dilute solution. The solvent is evaporated off, giving a 'pre-preg' which, if the resin is selected with this in mind) can be stored for several weeks.

The material can finally be moulded and cured under pressure in a mould, the best results being obtained when only a very slight excess of resin is present.

Figures quoted for thermoset resin composites, with different types of fibre, are, for flexural strength and flexural modulus respectively: asbestos/resin, up to 125 x 10^3psi and up to 14.5 x 10^6psi; carbon (type 1)/resin, up to 100 and up to 25; and silicon carbide/resin (which is still only in the early stages of development), 200-300 and 30-40.

Patents covering the ERDE fibre preparation processes are being filed and, if all goes well, licences will be made available to industry.

Applications for these composites could include rocket motor components such as cases, exhaust tubes and nozzles and airframe structures where light weight but high strength, high stiffness and possibly ablation resistance are needed. If the full properties of silicon carbide whiskers can be developed in resin composites, adds ERDE, they would give the highest properties, for the most exacting applications — but at correspondingly greater cost. A tentative price for silicon carbide whiskers is 20s to 40s a lb.

HIGH HEAT

Incorporation of asbestos fibres into rubber compounds is also being examined, but at present this appears to be of interest only in high heat-resistant synthetic rubbers.

In the rubber section at Waltham Abbey, however, an interesting piece of fall-out from military trouble-shooting is the development of a conductive natural rubber compound for anti-static purposes.

Designated ERDE natural rubber standard 1.7/70, the compound contains 70 parts of acetylene black per 100 of rubber. This was compared with a range of other rubbers containing the same loading of acetylene black and tested at a contact pressure of 15psi.

Results from this test gave an electrical resistance of only

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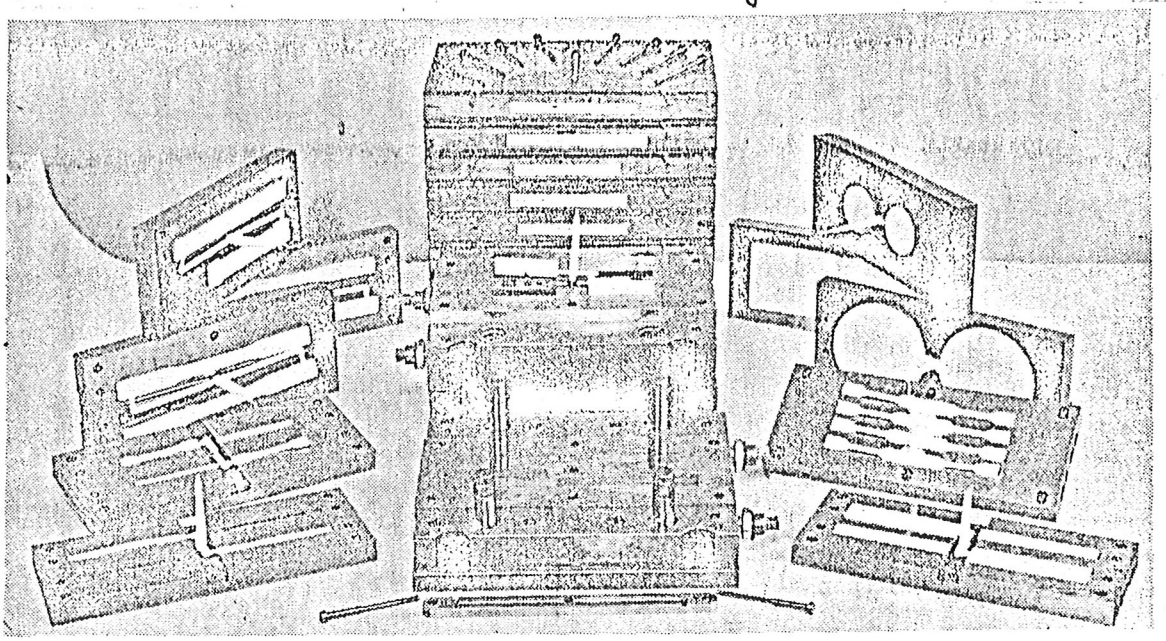

THESE are the low cost interchangeable cavity plate injection moulds for producing standard specimens which have been developed by the Explosives Research and Development Establishment. The bolster set and cavity plates were designed by ERDE's polymer development and application group.

6ohms for the ERDE NR compound, compared with 10^6ohms for a standard solid tyre rubber, 360ohms for Hypalon, 90ohms for a commercial conducting rubber, 30ohms for neoprene and 13ohms for butyl. Lower resistances were obtained from acrylate (4ohms), cis-1,4-polybutadiene (3), cis-polyisoprene (2.5), fluorinated rubber (0.6) and polysulphide (0.5).

Later tests with two special furnace blacks gave lower resistivities than acetylene black with similar loadings, but the incorporation of fibrous fillers such as carbon wool and aluminium staple gave rubbers of poor conductivity.

In the course of tests, the effect of repeated flexing under compression and under tension was studied, showing that after flexing for the equivalent of running a tyre for 1,000 miles the resistance of compound L7/70 was still well within the conducting range.

From tests on the Akron abrader and Goodrich flexometer, ERDE now concludes that the compound has better abrasion resistance and a lower heat build-up than either of the commercially available solid-tyre compounds, and is comparable with standard pneumatic tyre tread compounds. A full-scale evaluation in vehicle solid tyres is now in hand.

Also in hand in the rubber applications laboratory is a development programme on proofed fabrics. Initially directed towards the development of large flexible storage containers for liquids (in which a considerable amount of work was done on the design and strength of joints for Dracones and large pillow tanks), the programme is now aimed at obtaining basic engineering data on rubber-proofed nylon fabrics in an attempt to elucidate some of the failures that have occurred in Hovercraft skirts. Also under investigation are high-strength proofed fabric tapes for use in aircraft arrester gear.

Linked to both the plastics and rubber applications development is a polymer chemistry section, in which a novel method of tailor-making polymers to specific requirements by suitable selection of components is currently being developed.

LINKED

Generally, copolymers have chains consisting of two different monomer units linked together in random order. The relative proportions of the two components in the chains are fixed by the properties of the species involved and cannot be systematically varied over a wide range, but the new method, by judicious choice of ingredients, is claimed to enable varying proportions of the co-monomers to be incorporated.

The method relies on the reaction of a vinyl monomer with an alkali metal in the presence of a calculated amount of linking agent such as a dihalide or diepoxide. A strong cation solvating solvent such as tetrahydrofuran is necessary, as also is the absence of interfering species such as oxygen or water.

For example, copolymers of styrene and methylene in various predetermined ratios have been prepared by reacting styrene with lithium in the presence of the α,ω-dihalide Br $(CH_2)_n$Br, where n can be systematically varied. A dimerisation reaction occurs which appears to be followed almost invariably by a linking reaction, rather than a polymerisation step involving addition of more vinyl monomer.

This theory is supported by evidence from nuclear magnetic resonance which gives aliphatic to aromatic hydrogen ratios in close accord with those predicted by the theoretical equations.

A large number of regular copolymers has been prepared using monomers such as styrene, butadiene and isoprene, with many linking agents, and the section is currently evaluating what it believes to be a method with very wide synthetic possibilities, as it appears to make it possible to produce regular copolymers easily and in quantity. The fact that diene monomers can also be used suggests that rubbers can also be prepared with closely defined structures.

At the other end of the scale, for processing trials, ERDE has two smallish injection moulding machines which, as well as producing test pieces for evaluating the new composite thermoplastics, has produced some potentially useful fall-out in terms of tooling for injection moulding.

Like many another r and d department, ERDE has to watch the pennies, and this has given rise to investigation of ways to minimise the cost of tools.

One particular outcome has been the introduction of interchangeable mould cavities, by a rationalisation of the various moulding requirements of the department.

EVALUATION

To a certain extent, this experience may be less relevant to industry than some of the other work of ERDE, because the mouldings concerned are all similar and are produced not for commercial use but for evaluation purposes.

Thus it has proved possible to design cavity plates for some ten bar, disc, dumbell and plaque mouldings which can all be used in the same basic tool, while three types of gear wheel, 2¼in dia, 3½in dia, and the two sizes together can all be produced from the one basic mould.

Nevertheless, the fact that this has been done and the way it has been tackled may be of interest to many commercial moulders.

Of perhaps more general interest, however, is another cost-saving exercise in which ERDE has been working with steel-filled epoxy compounds to produce injection moulding tools for development runs.

Using this technique, the researchers have found that, for perhaps £20-£50 they can produce a development tool which would normally cost some pounds. The technique is familiar to patternmakers and most moulders, and the material currently being used is either formulated by Waltham Abbey or bought outside ready-made. An additional advantage, apart from the low initial cost, is that the tools made in epoxy/steel can readily be modified, by cutting and filling, allowing changes in component shape or gateing.

PROTOTYPE

ERDE reckons this technique (which has been worked out in consultation with a commercial moulder) is of general value for prototype tooling, allowing up to about 100 mouldings to be produced, before going to, say, Kirksite for semi-production runs and only then to the final steel tooling when all the bugs have been ironed out.

The two open days at Waltham Abbey were held in accordance with current Mintech policy of strengthening its contacts with industry and showing industry as much of its work as it can.

This aspect was stressed by the director of ERDE, Dr L. J. Bellamy, who pointed out the facilities at Waltham Abbey which are available to industry for general advice and consultation, or specific studies and development projects, either free of charge or on a fee-paying basis, as appropriate, subject to the availability of capacity.

'After all', he said, 'it's the first time in 400 years that we've let outsiders in' — and one hopes they won't leave it another 400!