4. G. MENGES and E. ALF, *J. Elast. Plast.* 7 (1975) 47.
5. R. J. CRAWFORD and P. P. BENHAM, *Polymer* 16 (1975) 908.
6. J. M. SCHULTZ, "Treatise on Material Science and Technology" Vol. 10, Part B (Academic Press, New York and London, 1977) p. 599.
7. F. X. de CHARENTENAY, F. LAGHOUATI and J. DEWAS, 4th International Conference on Defects, Yield and Fracture of Polymers, Cambridge, March 1979, Paper 6.

Received 3 February
and accepted 27 February 1981

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Oxidation behaviour of aluminium alloys containing magnesium and lithium

The thermal oxidation of certain Al alloys has been studied by new methods [1, 2] in the last few years. There is an increasing interest in understanding the structural and compositional changes occurring at metal–gas-phase boundaries. Heat-treatment is one of the most common technological procedures used in aluminium semi-fabrication. Depending on the composition of the bulk material, compositional and structural changes may occur at the metal–gas-phase boundary.

The question investigated by electron microscopy and thermogravimetry was: how is the oxi dation behaviour of Mg-containing alloys is influenced by Li?

The compositions of the alloys examined are given in Table I. The alloys were prepared from high purity base materials. The homogenized alloys were rolled to 0.5 mm and for electron microscopy purposes the samples were solution heat-treated and then cold-water quenched before electropolishing. *In situ* oxidation experiments were conducted in an AEI EM 7 high voltage electron microscope (HVEM) using 500 kV and a vacuum of 2×10^{-4} Pa at 500° C for 0.5 h. The heating rate used was about 100° C min^{-1}. Other samples prepared for electron microscopy were heat-treated at 10^5 Pa in air at 500° C for 4 h. Morphological and structural observations were evaluated and compared to the results obtained by thermogravimetric (TG) measurements. The TG measurements were made in the low-temperature furnace of a Mettler thermobalance (No 212). The surface area of the samples was 42 cm^2. The material was heat-treated in a dried air flow of 16 l h^{-1} at 350° C

TABLE I Composition of the alloys in wt%

Alloy	Zn	Mg	Li
Al–Zn–Mg	5.0	2.1	–
Al–Zn–Mg + Li	5.0	2.1	0.19

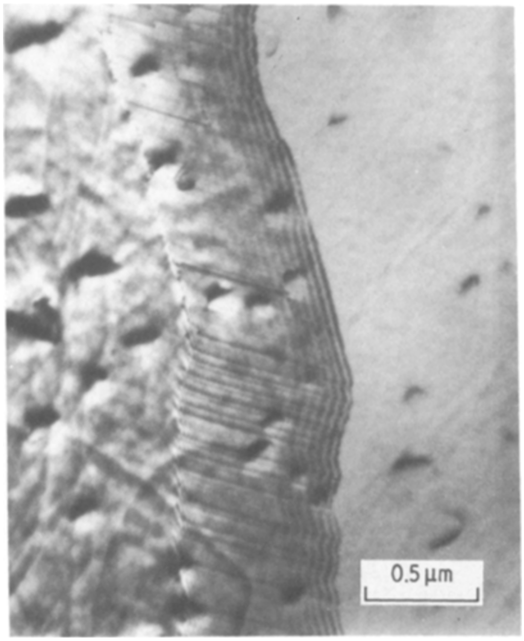

Figure 1 Oxide crystals on the surface of an Al–Zn–Mg alloy heat-treated at 500° C for 1 h in air at 2×10^{-4} Pa.

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TABLE II Summary of direct observations of oxidation at 2×10^{-4} Pa and 500° C for 0.5 h

Alloy	Temperature of crystal nucleation (°C)	Average diameter of primary crystals (nm)	Density of primary crystals (cm^{-2})	Structure of crystals
Al–Zn–Mg	500	~ 100	5×10^8	$MgAl_2O_4$ MgO
Al–Zn–Mg + Li	360	~ 30	2.5×10^{11}	Li_2O MgO

for 1 h and then at 500° C for 2.5 h with a heating rate of 25° C min^{-1}.

The results of *in situ* experiments are shown in Table II. The crystals which developed on the surfaces of Al–Zn–Mg and Al–Zn–Mg–Li after the vacuum heat-treatment can be seen in Figs 1 and 2, respectively. In the case of Al–Zn–Mg–Li, rapid crystallization occurs due to Li enriched in the amorphous oxide layer [3]. This is shown by a high density and low average diameter of the crystals. The results of heat-treatment in air are shown in Table III. Figs 3 and 4 show crystals which developed in the two alloys after the heat-treatment in air. In the case of heat-treatment in air at 1 atm a much higher crystal growth rate could be observed on the Li-containing material.

TABLE III Summary of observations of oxidation at 10^5 Pa and 500° C for 4 h

Alloy	Average diameter of primary crystals (nm)	Structure of crystals
Al–Zn–Mg	~ 51	$MgAl_2O_4$
Al–Zn–Mg + Li	~ 146	$LiAl_5O_8$ MgO

Figs 5 and 6 show the results of oxygen uptake in air at 10^5 Pa (1 atm). The very high oxygen uptake of the lithium-containing alloy is explained by the oxide structures identified by electron diffraction analysis (see Table II). Oxygen uptake is considerably increased by Li.

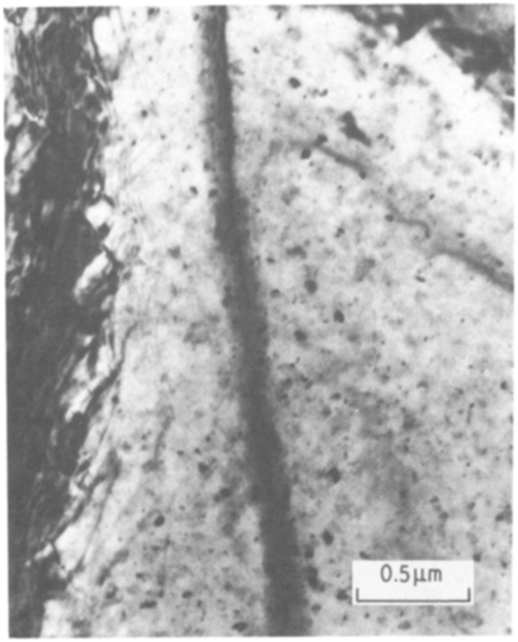

Figure 2 Oxide crystals on the surface of an Al–Zn–Mg–Li alloy heat-treated at 500° C for 1 h in air at 2×10^{-4} Pa.

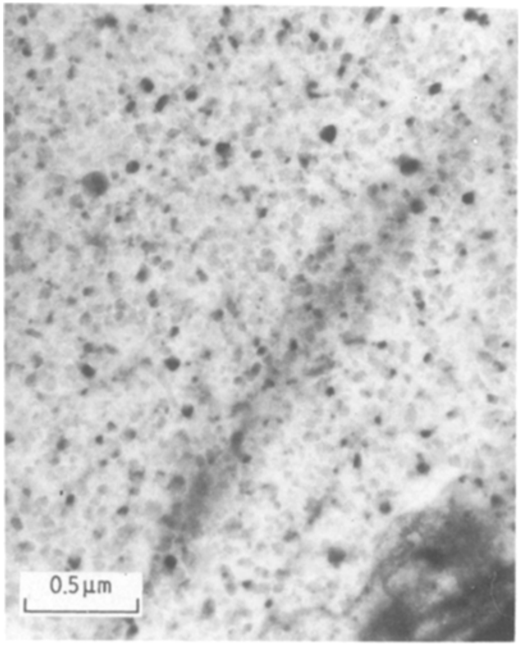

Figure 3 Oxide crystals on the surface of an Al–Zn–Mg alloy heat-treated at 500° C for 4 h in air at 10^5 Pa.

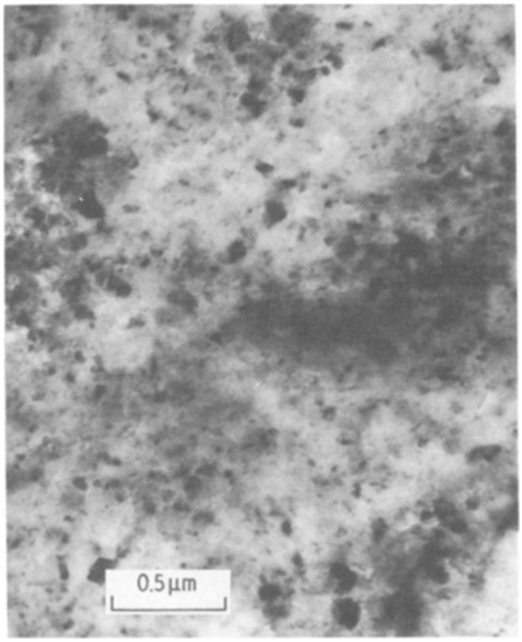

Figure 4 Oxide crystals on the surface of an Al–Zn–Mg–Li alloy heat-treated at 500° C for 4 h in air at 10^5 Pa.

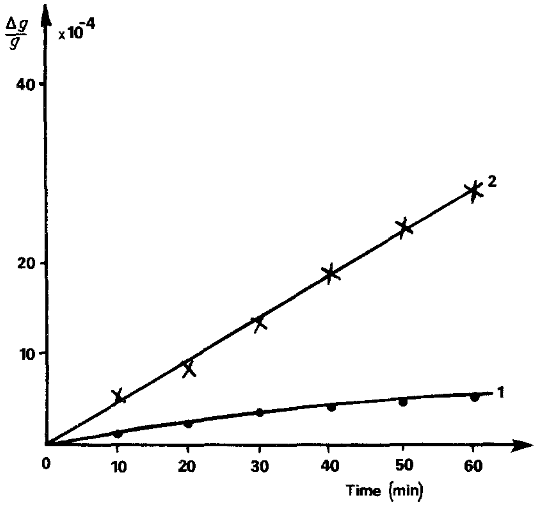

Figure 5 Oxygen uptake by (1) Al–Zn–Mg and (2) Al–Zn–Mg–Li alloys, heat-treated at 350° C for 1 h in air at 10^5 Pa.

Acknowledgements

One of the authors (Á. Csanády) is grateful for the use of the HVEM given by the Max Planck Institut für Metallforschung, Stuttgart and especially thanks to Professor A. Seeger and to DAAD for the scholarship. The authors wish to thank Dr T. Turmezey for the alloys.

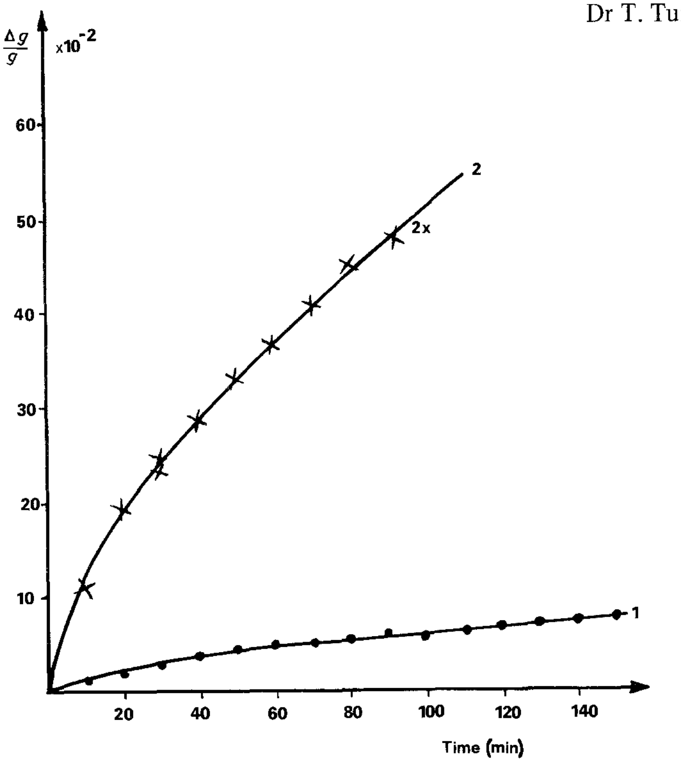

Figure 6 Oxygen uptake by (1) Al–Zn–Mg and (2) Al–Zn–Mg–Li alloys, heat-treated at 500° C for 2.5 h in air at 10^5 Pa.

References

1 G. M. SCAMANS, E. P. BUTLER, *Met. Trans.* 6A (1975) 2055.

2. F. DEGREVE, R. FIGARET, P. LATY, *Int. J. Mass Spectr. Ion Phys.* 29 (1979) 351.

3. Á. CSANÁDY, Proceedings of the 7th International Light Metal Congress, June 1981.

Received 4 February
and accepted 27 February 1981

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Direct lattice resolution in polydiacetylene single crystals

With the advent of modern electron microscopes the direct observation of structure in crystals of metals and ceramics by lattice imaging [1] is now becoming a relatively routine matter. The technique of using high resolution electron microscopy (HREM) to directly resolve lattice planes in crystals was first demonstrated in a classical series of papers by Menter [2, 3]. He showed that images could be obtained from thin crystals of copper and platinum phthalocyanines which contained fringes of the same separation as the lattice planes in these materials. A major drawback in extending the technique to other materials such as polymer crystals is that the crystals of organic materials invariably undergo radiation damage in the electron beam. The problem of beam damage is crucial in controlling the application of HREM to polymer crystals since it causes a rapid deterioration in the potential resolution [4]. Another problem that is encountered when studying polymers by electron microscopy is specimen preparation and the conventional techniques used include the precipitation of chain-folded lamellar crystals from dilute solution or microtoming or cleaving thin sections from bulk samples. Neither of these techniques are completely satisfactory as solution-grown crystals can only be observed in directions parallel to chain axis and microtoming or cleavage can introduce artefacts and cause damage to the crystals. Nevertheless, a careful and elegant study has been recently made by Dobb and co-workers [5, 6] upon the structure of fragments obtained from high-modulus aromatic fibres in which they were able to resolve lattice fringes as small as 0.43 nm from microcrystalline areas.

A significant step forward in the preparation of polymer crystals in morphologies suitable for study by electron microscopy was made with the use of solid-state polymerized polydiacetylene single crystals [7]. These materials developed about 10 years ago by Wegner and co-workers [8] are made by the polymerization of suitable substituted diacetylene monomer single crystals. Because the polymers are polymerized in the form of single crystals in the solid state the polymer single crystals can be 100% crystalline, of macroscopic dimensions and contain molecules in chain-extended conformations [7]. There is currently considerable interest in studies of the structure and properties of polydiacetylene crystals because of their interesting and unusual optical, electronic and mechanical properties. Many of these properties reflect the single-crystal nature of the polymers and it is the purpose of this communication to show briefly how the fundamental crystal lattice can be observed directly by HREM.

Recent studies by the authors and co-workers [7, 8, 10] have demonstrated the usefulness of transmission electron microscopy in studying the structure of polydiacetylene single crystals. Previous work has concentrated upon crystals of the p-toluene sulphonate derivative (pTS) [11]. It has been shown that these crystals contain stacking faults [10] and dislocations [12] and undergo twinning upon deformation [9]. Single crystals of pTS can be obtained in the form of thin (50 to 200 nm thick) foils which are relatively resistant to radiation damage in the electron beam. HREM has has been attempted with pTS but attempts to image the crystal lattice have met with only limited success. It is thought that this may be because either the crystals suffer from radiation damage at the high magnification (~ x 100 000)

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