observation of the 52% γ-Fe–23.19 wt% Cr–4.91 wt% Ni–0.025 wt% C alloy.

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Chemical polishes for $\{100\}$, $\{110\}$ and $\{111\}$ MgO surfaces

Stokes *et al.* [1] discovered almost 20 years ago that $\{100\}$ MgO single crystal surfaces can be chemically polished by immersion for 1 min in a boiling 85 wt% solution of H_3PO_4 in H_2O. Since then many investigators have published claims for the efficacy of variants of this procedure, often without supplying sufficient experimental detail to enable others to reproduce their results. For example, Stokes [2] recommended using 4 parts of H_3PO_4 (concentration unspecified) diluted with 1 part of H_2O, while Groves [3] suggested adding 2 to 3 wt% of either concentrated H_2SO_4 or concentrated HNO_3 to a 67 wt% solution of H_3PO_4. Similarly, Ghosh and Clarke [4] found that an 88 wt% solution of H_3PO_4 caused etching rather than polishing at temperatures less than 125° C, whereas Elkington *et al.* [5] and Ogawa [6] reported polishing satisfactorily in H_3PO_4 solutions of unspecified concentration at 110° C and 120° C, respectively.

In addition, Ogawa [6] has succeeded in using a jet polishing method to chemically thin $\{110\}$ oriented MgO slices in H_3PO_4 of unspecified concentration at 120° C; and Rice [7] has polished not only $\{100\}$, $\{110\}$ and $\{111\}$ oriented single crystal surfaces, but also the surfaces of polycrystalline samples, in boiling H_3PO_4 solutions, although he gives no details of the precise experimental conditions he employed.

In an attempt to remedy this situation, the present authors have made a detailed study of the conditions leading to the optimum chemical polishing of $\{100\}$, $\{110\}$ and $\{111\}$ surfaces of MgO single crystals. Spectrochemical analyses of these crystals are reported in Table I, and show the impurity content to vary considerably from crystal to crystal.

All specimens took the form of flat slabs ~ 1 cm × 1 cm × 0.5 cm in size. Those having their largest surfaces parallel to $\{100\}$ were shaped by cleavage, and those oriented parallel to $\{110\}$ and $\{111\}$ were cut to shape with a water-cooled diamond saw. Then, because the impact studies for which these specimens were intended required flat rather than slightly undulating surfaces, all slabs were polished mechanically, first on wet SiC papers down to 600 grit, and then with Al_2O_3 slurries of down to 0.05 μm particle size. This procedure reduced the amount of damaged material needing to be removed chemically, and hence reduced the polishing time and the opportunity for undulations to develop. It was not, however, a necessary prerequisite for successful chemical polishing, for other experiments showed that equally damage-free – though slightly undulating – surfaces

TABLE I Spectrochemical analyses of specimens

Element	Concentration (p.p.m.)
Ca	100–200
Al	40–100
Mn	50
Fe	100–200
Si	50
Ti	20–50

No trace was found of Sr, Ba, Be, Cu, Ag, B, Cr, V, Ni, Mo, Pb, Bi, Ge, In or Zr.

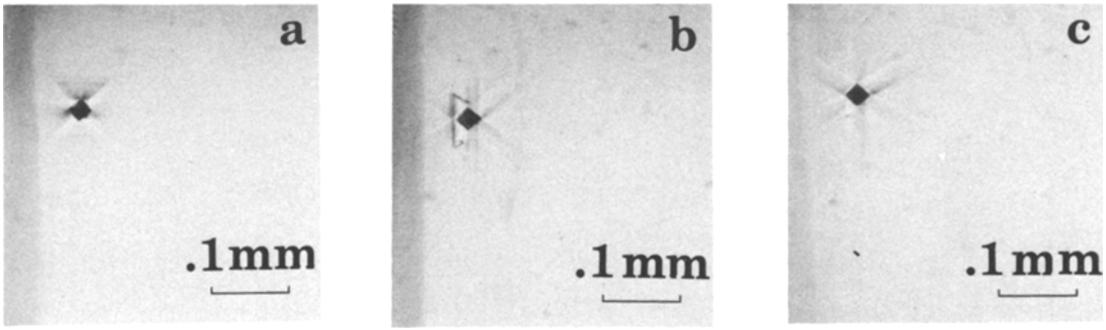

Figure 1 Chemically polished surfaces of MgO single crystals. The slip lines around the hardness impressions confirm the orientations as {1 0 0}, {1 1 0} and {1 1 1} in (a), (b) and (c), respectively.

could be produced from both cleaved {1 0 0} slabs and rough-cut {1 1 0} and {1 1 1} slabs merely by increasing the (chemical) polishing time.

Actual chemical polishing was carried out with the specimen sitting in a shallow Teflon tray on the bottom of a large Pyrex beaker containing a considerable volume (~ 200 cm^3) of reagent grade H_3PO_4. The tray served to prevent the specimen sticking to the glass, and the large volume of H_3PO_4 (i) ensured temperature stability (particularly during the frequent replacements of small amounts of evaporated H_2O) and (ii) to reduce the changes in composition resulting from evaporation of H_2O and/or dissolution of MgO. Porcelain "bumping stones" were used to control delayed boiling and promote what was, in effect, uniform stirring whenever boiling occurred; and the polishing solutions were invariably discarded as soon as visible discoloration occurred. After polishing, specimens were rinsed successively in boiling H_2O, cold H_2O and pure ethanol, and then dried in a stream of warm air.

TABLE II Optimum chemical polishing conditions

Surface orientation	Polishing conditions
{1 0 0}	7–10 min immersion at 120° C in gently boiling 67 wt% H_3PO_4 containing 0.2–0.3 wt% concentrated H_2SO_4 or HNO_3
{1 1 0}	20–30 min immersion at 130° C in gently boiling 73 wt% H_3PO_4 containing 0.2–0.3 wt% concentrated H_2SO_4 or HNO_3
{1 1 1}	20–30 min immersion at 159° C in gently boiling 85 wt% H_3PO_4 containing 0.2–0.3 wt% concentrated H_2SO_4

With these precautions, it was found that the optimum polishing conditions were those listed in Table II.

Figs. 1a to c, respectively, show {1 0 0}, {1 1 0} and {1 1 1} surfaces polished under these conditions. The Vickers microhardness impressions and associated slip traces shown in each figure serve merely to confirm the surface orientation and ensure proper focusing of an otherwise featureless image. No effects of impurity content on polishing behaviour could be discerned in any case. There was, however, some tendency for the {1 1 1} specimens to fail by thermal shock when they were first placed into the hot H_3PO_4.

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