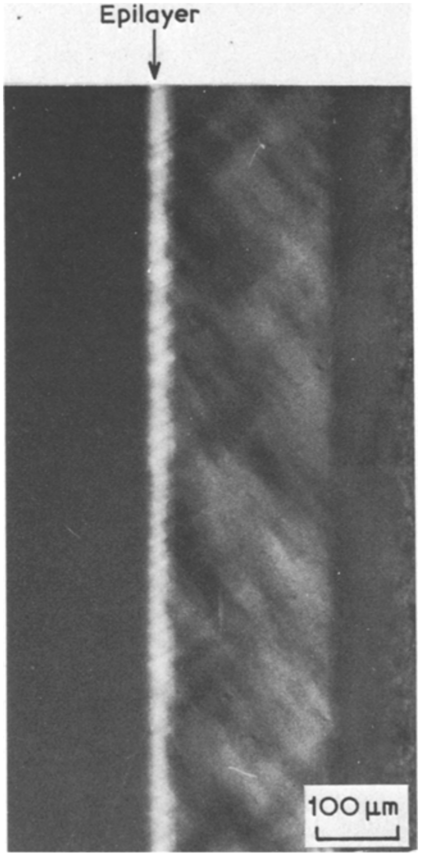

Figure 6 Bi-refringent transmission through an as-grown VPE sample.

Thus we have found that stress-induced bi-refringence provided the same information on the plastic deformation in GaP as revealed by SEM cathodoluminescence showing that the polarizing microscope provides a much more convenient and inexpensive substitute for the SEM for this purpose. Additional information has been obtained on the as-grown material. The sharp features observed at the epilayer–substrate interface and the accompanying strain may account for the sharp drop in the minority carrier lifetime at the interface observed by Blenkinsop *et al.* [8].

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M. ZAFAR IQBAL
Department of Physics,
Quaid-i-Azam University,
Islamabad, Pakistan

Selected-area diffraction ring patterns in Al–Zn–Mg powders

Thin-foil observations of as-produced pre-alloyed aluminium powders have raised the question as to the nature of the microstructure of these powders [1–5]. The interpretation of selected-area electron-diffraction ring patterns ranges from the presence of non-crystalline phases [1, 4] to either an amorphous carbon artifact introduced during the splat-sequence process or an oxide formation introduced during ion-beam thinning [3]. The purpose of this letter is to report on a different type of artefact discovered while attempting to prepare thin foils of an as-produced Al–Zn–Mg* alloy manufactured by four different commercial vendors. Based upon average dendrite arm-spacing measurements [6] (see Fig. 1), the solidification rates varied between 10^4 to 10^5 K sec^{-1} [7]. As mentioned, these cooling rates are five orders of magnitude slower than those calculated for the powders where an amorphous structure was reported [1, 4].

TEM thin foils were initially prepared in a

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*Composition: 6.5 wt% Zn, 2.5 wt% Mg, 1.5 wt% Cu, 0.40 wt% Co, balance Al.

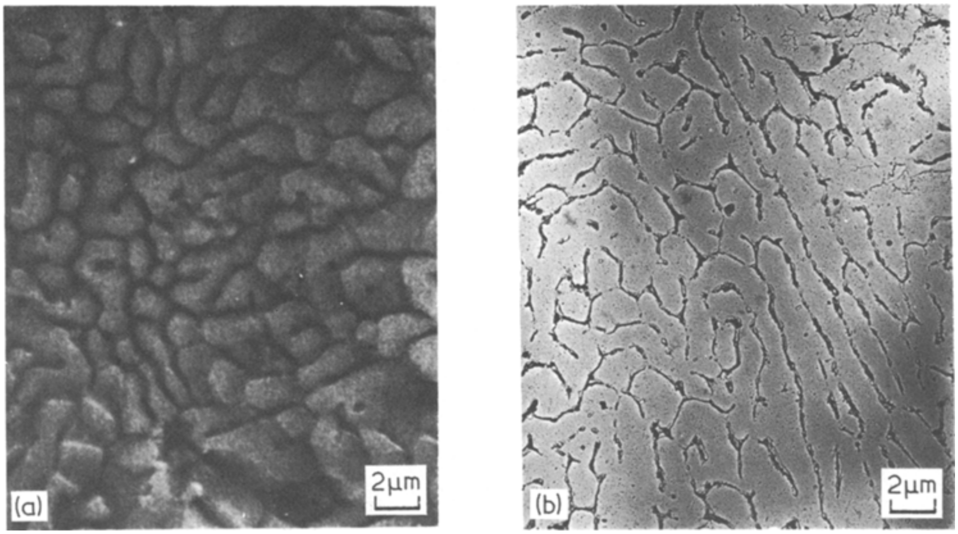

Figure 1 Metallographic polished and etched (Keller's) cross-section of a typical powder: (a) SEM photograph and (b) TEM replica.

commercial ion-milling unit using 98.5% pure (oil-pumped) argon. Fig. 2a shows the complete absence of the structure shown in Fig. 1. Moreover, SAD in the outlined area produced a continuous three ring pattern as shown in Fig. 2b which was identified as gamma Al_2O_3, suggesting that the oxygen partial pressure was sufficiently high to build continuously an amorphous layer on the freshly exposed aluminium surface. Thus, the experimental apparatus was modified by placing an oxygen trap (consisting of titanium sponge heated to 600° C) between the argon tank and the ion guns. Once again, continuous ring patterns were observed; however, all of these rings were too diffuse for identification with any certainty, and the TEM grid was placed in an electron microprobe for further analysis. Fig. 3a shows the appearance of a spherical particle which was milled flat by the ion plasma, and Fig. 3b is a chlorine map of the surface. These results suggest that the chlorine contained in the titanium sponge was released in gaseous form and reacted with the freshly exposed aluminium surface to form $AlCl_3$. For subsequent ion-thinning runs, both the oxygen

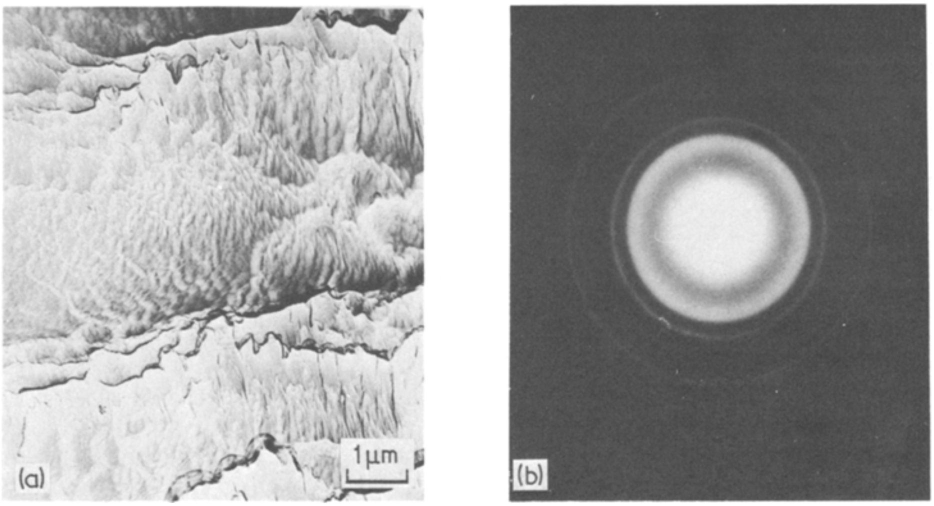

Figure 2 Thin foil prepared in a commercial ion-milling unit using 98.5% pure argon: (a) bright-field, and (b) selected-area diffraction from this area.

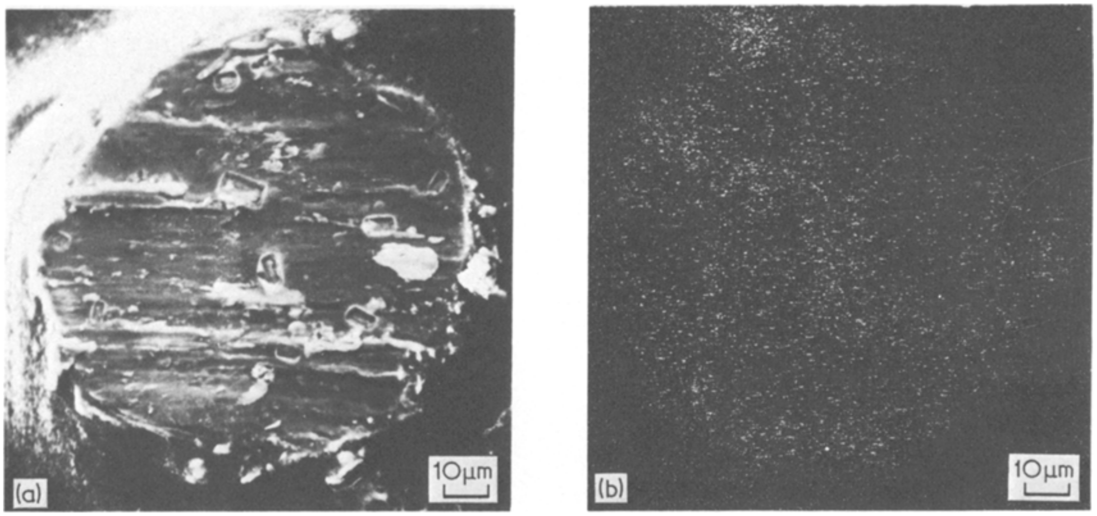

Figure 3 Chlorine contamination of a powder using 98.5% pure argon which was passed over 600° C titamium sponge prior to thinning: (a) SEM photomicrograph of ion-milled surface, and (b) chlorine map.

trap and the 98.5% pure argon tank were replaced with a tank of 99.999% pure argon.

Thin foils prepared after these changes again showed such diffuse ring patterns that they could neither be unequivocally identified nor quanatively compared with the previous diffused ring pattern discussed above. An energy-dispersive analysis performed in an electron microprobe showed the presence of chromium, iron, and nickel. Since the specimen holder for the commercial ion-milling unit is made of stainless steel, these facts suggested that the specimen holder was being eroded as well

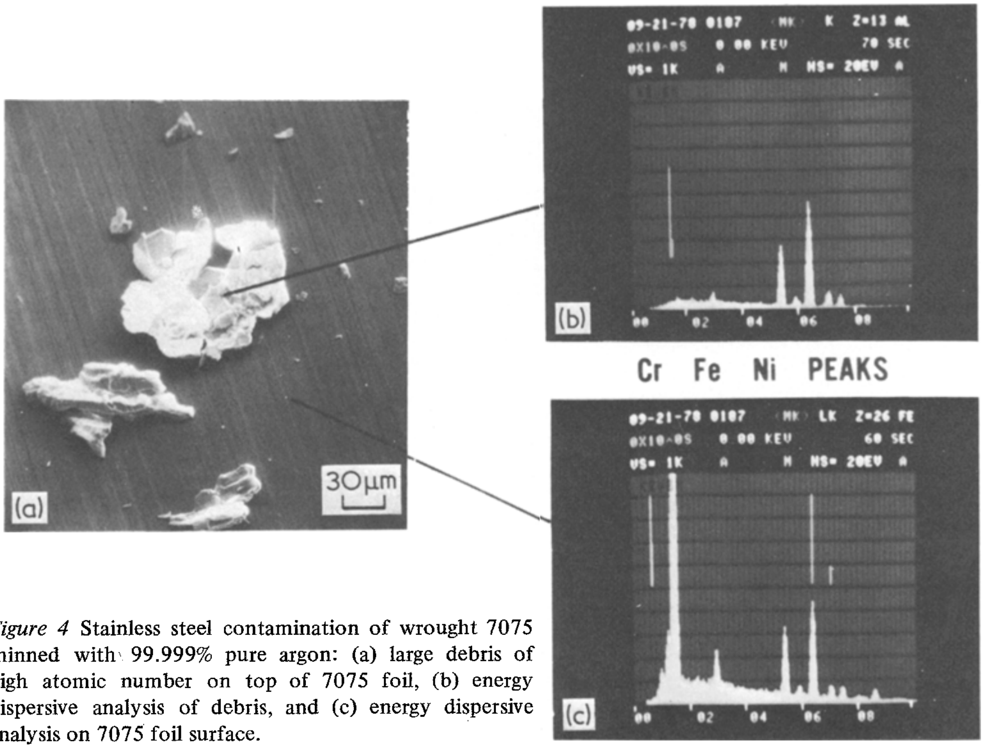

Figure 4 Stainless steel contamination of wrought 7075 thinned with 99.999% pure argon: (a) large debris of high atomic number on top of 7075 foil, (b) energy dispersive analysis of debris, and (c) energy dispersive analysis on 7075 foil surface.

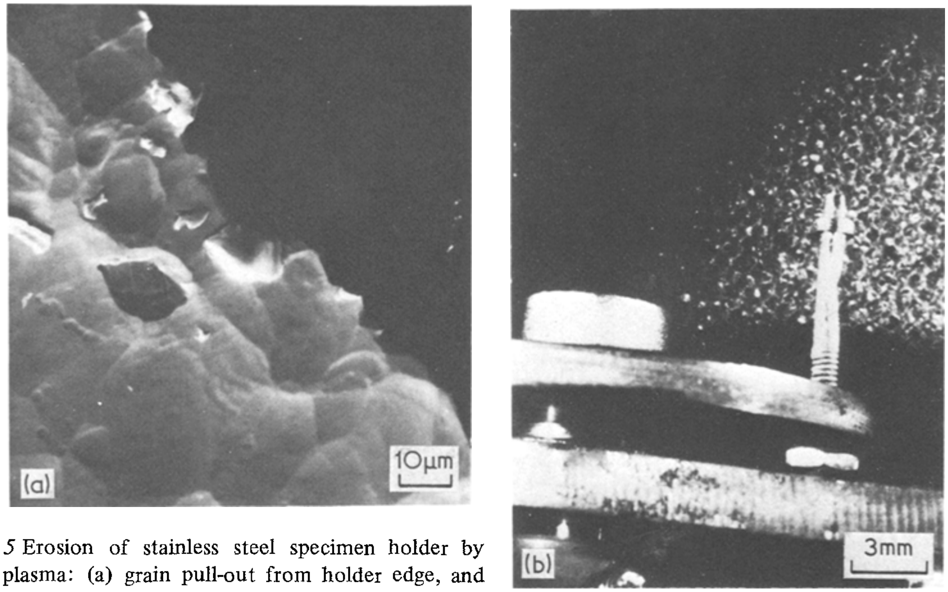

Figure 5 Erosion of stainless steel specimen holder by argon plasma: (a) grain pull-out from holder edge, and (b) support pin erosion.

as the powder sample of interest. To verify this hypothesis, a thin wafer of wrought 7075* (which has the same nominal composition of the powders less cobalt) was thinned next in the commercial ion-milling unit and then removed for examination in the electron microprobe.

Fig. 4a illustrates the surface condition of the 7075 wafer on which copious amounts of large (50 to 100 μm) debris of high atomic number are present, as indicated by the high contrast relative to the aluminium. Spectrographic analysis of the debris confirms the presence of chromium, iron, and nickel. However; it is even more curious to observe the presence of these same three elements on the 7075 wafer away from the debris. These results are taken to mean that there are two kinds of stainless-steel contamination. First, the edge of the stainless steel specimen holder is being eroded away from the argon plasma and large portions fall onto the specimen during its rotation in the ion-milling machine. Fig. 5a gives evidence to support this interpretation. Second, the ion-milling machine specimen holder is completely shrouded by the argon plasma. The atoms from the stainless steel are removed from the holder, but they are too heavy to be completely removed by the argon plasma charged at the relatively low accelerating voltage used for thinning aluminium-based alloys. Figs. 4c and 5b contain evidence to support this interpretation.

If the argon plasma does not contain sufficient kinetic energy for removing the atoms from the stainless steel during thinning of aluminium alloys, there appear to be three possible solutions. First, increase the accelerating voltage of the argon plasma to completely remove the stainless steel atoms and risk deep ion implantation of argon inside the foils. Maintain the same voltage as before, but place a sacrificial coating of a light atomic element (e.g. carbon) over the specimen holder which will, hopefully, be carried clear of the specimen area. Third, fabricate a specimen holder from a machinable ceramic which is virtually unaffected by the argon plasma. These experiments are now in progress.

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*Nominal composition: 6.5 wt% Zn, 2.5 wt% Mg, 1.5 wt% Cu, balance Al.

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R. E. OMLOR
Systems Research Laboratories Inc,
2800 Indian Ripple Road,
Dayton, Ohio 45440,
USA

J. S. SANTNER*
Metals and Ceramics Division,
Air Force Materials Laboratory,
Wright-Patterson Air Force Base,
Ohio 45433, USA

---

*Present address: Inland Steel Company Research Laboratories, 3001 East Columbus Drive, East Chicago, Indiana 46312, USA.

Indexed X-ray diffraction data for the sialon X-phase

The structure and composition of the X-phase of the sialon system have been subjects of controversy [1–4]. Recently, conclusive electron diffraction data proved that the structure is triclinic, with $a = 0.856$ nm, $b = 0.985$ nm, $c = 0.969$ nm, $\alpha = 70°$, $\beta = 81°$, $\gamma = 81°$. This cell could also be "reduced" to: $a = 1.194$ nm, $b = 0.9685$ nm, $c = 0.8542$ nm, $\alpha = 99.2°$, $\beta = 90°$, $\gamma = 124.3°$, with no increase, however, in the symmetry of the structure [5]. The composition, determined with the aid of experiments at various high temperatures [2] and through tie-line determinations [6], lies close to $Si_{12}Al_{18}O_{39}N_8$ [6], $Si_3Al_6O_{12}N_2$ [7] or $Si_2Al_3O_7N$ [8].

Since large enough single crystals of the phase are not available, the structure determination must be based on X-ray powder diffraction data, which, in this complicated multi-component structure, are far from being sufficient. The complexity is further increased by the difficulty in obtaining the pure phase and by the extremely finely spaced planar defects in the structure, which can give rise to intensity variations.

In view of our present knowledge of the structure, it was observed [5] that the existing X-ray powder diffraction data, as reported in several publications, included reflections which do not belong to the structure, and, more often, lacked many of the important reflections which do belong to the X-ray diffractogram. We present here a fully indexed powder data with the observed relative intensities, which can serve for detecting the X-phase and its relative amount in multi-phase samples.

The mixed powder samples, with compositions shown in Table I, were hot-pressed at 1740 to 1760° C under 30 MPa for 120 min. The final samples were estimated to contain at least 95% of the X-phase and less than 2% of other crystalline phases.

The samples were examined as-hot-pressed (as-polished bulk polycrystals) in an X-ray diffractometer with a LiF monochromator, and in powdered form in a monochromated Guinier camera, with Cu*Kα* radiation in both cases. The relative intensities were determined visually.

Table II lists the indexed observed diffraction lines with their observed intensities. The low symmetry and the relatively large unit cell give rise to numerous reflections; 145 planes with lattice

TABLE I The starting material

Sample	Composition (wt %)			
	Si_3N_4	SiO_2	Al_2O_3	AlN
A–1	17.7	31.7	50.6	–
A–2	–	54.5	24.7	20.8

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