

conditions such as those used here.

The remaining reaction mixture was treated with enough acetone to cause precipitation of an inorganic salt. This was separated by filtration and the filtrate was vacuum distilled to remove excess solvent. The purple viscous residue was chromatographed on alumina using a sequence of eluants of increasing polarity: cyclohexane, benzene, benzyl alcohol, isopropanol, ethanol, and water. Reaction mixture components were found in all but the cyclohexane elution. The elution bands were subjected to infra-red and nmr spectroscopy and to contact with picric acid (in an attempt to form an identifiable picrate): no *p*-phenylene diamine or aromatic nitrates were detected.

To support the presence of a *p*-phenylene diamine condensation partner and to remove the possibility of any *m*-phenylene diamine partner, pure samples of the two diamines were separately subjected to exactly the same reaction conditions as the aromatic amine fibre. Then, each reaction mixture was treated similarly to fibre reaction mixture. Following this, infra-red spectra were taken of each of the bands eluted by column chromatography of the two reaction mixtures. The spectra derived from the *m*-phenylene diamine and *p*-phenylene diamine reactions were then compared with those from the fibre reaction. Comparison showed that the Kevlar and *p*-phenylene diamine reaction mixtures contained common

components, and that neither mixture had anything in common with the *m*-phenylene diamine reaction mixture. These results and the fact that *m*-phenylene diamine as a fibre building block would be unlikely in view of the high modulus of the fibre reinforced the choice of *p*-phenylene diamine as partner to terephthalic acid.

In conclusion, the high modulus organic fibre, Kevlar 49, when broken down chemically in our laboratory yielded terephthalic acid. From this result and from further analysis of the reaction mixture, we have assigned equimolar amounts of terephthalic acid and *p*-phenylene diamine as building blocks of the fibre.

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Additional observations on the strength/nitrided density relationship for a reaction sintered silicon nitride

In a recent publication [1] we demonstrated the existence of a linear relationship between mean strength and nitrided density for compacts prepared from a particular silicon powder, P (mean particle size 25 μm , maximum particle size 75 μm , specific surface area 0.7 m^2g^{-1}) nitrided to weight gains of less than 60%. These data were accumulated over a period of several months from experiments performed in a particular nitriding furnace. Some degree of consistency of the furnace contents (type and number of silicon compacts) was maintained throughout that series of experiments which is designated group I.

We have since performed additional nitriding experiments in the same furnace. Some of these experiments (designated group II) included silicon compacts made from the same powder batch (powder P) as that previously reported [1] as well as compacts prepared from other finer particle size silicon powders [2]. During these additional nitriding experiments (group II) the furnace contents were nominally identical from one experiment to the next but were significantly different from the furnace contents of the experiments of group I. Other nitriding experiments which included silicon compacts prepared from powder P have been conducted in this same furnace, but the contents of the furnace varied from one experiment to the next and therefore this group of experiments is not strictly comparable to group I

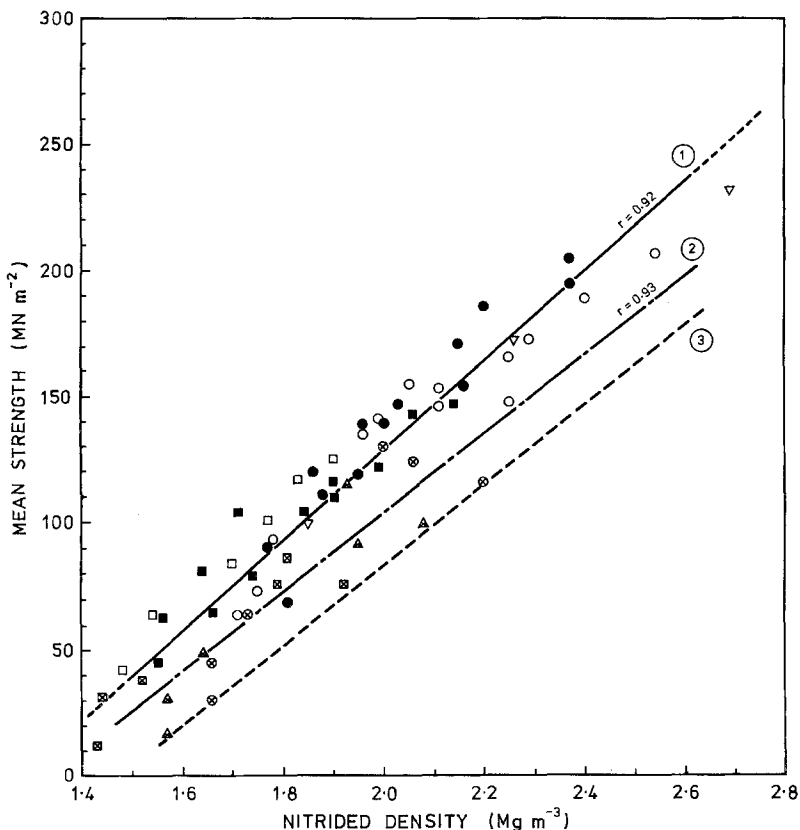


Figure 1 Mean strength versus nitrided density for compacts isostatically pressed from powder P and argon-sintered for 5 h at 1175° C.

Group I [1] ● pressed 185 MN m⁻², ■ pressed 31 MN m⁻². Group II [2] ⊗ pressed 185 MN m⁻², ▲ pressed 92 MN m⁻², ⊠ pressed 31 MN m⁻². Group III ▽ pressed 400 MN m⁻², ○ pressed 185 MN m⁻², □ pressed 31 MN m⁻². Line 1 ——— least squares fit for all data, line 2 - - - - least squares fit for group II data, line 3 ······ estimated lower bound of data.

or group II experiments. These additional experiments, although not representing a consistent series, are designated group III. The experimental details of all experiments were the same as those previously reported [1] except that additional silicon compacts were isostatically pressed at 92 MN m⁻² (group II) and 400 MN m⁻² (group III). The resulting silicon compact densities for powder P isostatically pressed at 31, 92, 185 and 400 MN m⁻² and heated for 5 h in argon at 1175° C were 1.34, 1.46, 1.55 and 1.66 Mg m⁻³ respectively.

The values of strength and nitrided density for powder P compacts from groups I, II and III are plotted in Fig. 1. The closed circles and closed squares represent group I data [1], the open symbols containing a cross are the data from group II [2] and the uncrossed open symbols are data

from group III experiments. In Fig. 1, line 1 represents a least squares fit to all the data (groups I, II and III). The equation of this line,

$$\sigma = 178 \rho_n - 228$$

is very similar to that for group I alone [1]:

$$\sigma = 181 \rho_n - 227$$

where σ is mean strength in MN m⁻² and ρ_n is the nitrided density in Mg m⁻³. The correlation coefficients for the two sets of data are very similar (0.92 and 0.96 respectively).

Line 2 Fig. 1 represents the least squares fit for powder P compacts from group II experiments: the corresponding equation is

$$\sigma = 155 \rho_n - 206.$$

The reasons for the differences between lines 1 and 2 are unknown at present. The major known difference between group I and group II experiments is the inclusion of compacts prepared from fine silicon powders in group II experiments and the influence of one type of silicon powder compact upon another during nitriding may be important.

The fact that we have observed differences between groups of experiments is of importance and indicates that caution must be exercised when comparing data obtained from different groups of experiments. The inclusion of control samples in comparative experiments appears necessary, although even this approach may not be entirely satisfactory due to the possibility of interaction between experimental and control materials.

In our earlier paper [1] we discussed the observation that compacts of powder P nitrided to weight gains greater than 60% frequently have mean strengths which fall below the values expected from the strength/density relationship. This observation was based on data accumulated at this laboratory from experiments in different furnaces, using various nitriding schedules and variable furnace contents. We suggested [1] that the explanation for this might be the existence of a flaw other than the largest effective pore which becomes strength controlling at higher weight gains when the pores are becoming small. Whilst such an explanation cannot be disregarded, attention is drawn to line 3 in Fig. 1 which represents the lower bound of the data. Lower than anticipated strength values are most probably the consequence

of a change in the previously derived strength/density relationship [1] caused by as yet unidentified variable parameters in the nitriding process. These low values are probably related to another strength/density relationship pertinent to the particular conditions existing during the experiments concerned.

It is interesting to speculate that if the variation of unidentified parameters can lead to a lower strength/density relationship than that recorded for group I experiments [1], then other conditions may be attainable when a higher strength/density relationship applies. Consequently, identification of the parameters which cause the observed changes in the relationship could lead to the development of stronger reaction sintered silicon nitride.

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Plastic deformation of $Ni_{0.66}Fe_{2.34}O_4$ spinel ferrite single crystals

Plastic deformation of oxides with the spinel structure has so far been studied through the example of $(Al_2O_3)_n MgO$. Some consistent results begin to appear [1, 2]. It is for example well accepted that glide directions are $\langle 110 \rangle$ and that glide planes depend on the stoichiometric ratio n ; glide on $\{111\}$ planes seems to occur for stoichiometries $n = 1$ and $n = 3$ whereas $\{110\}$ glide planes are activated for $n \sim 2$ [3–5]. In the present note, preliminary results are given of an

extensive investigation of the mechanical properties of an alternative spinel compound $NiFe_2O_4$.

Single crystals of $Ni_{0.66}Fe_{2.34}O_4$ ($8 \times 3 \times 3 \text{ mm}^3$) with $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ large axes have been strained between 1190 and 1516°C (0.73 to $0.9 T_M$) in compression under constant strain-rate ($\sim 2.5 \times 10^{-4} \text{ sec}^{-1}$). After deformation, slip lines were observed either directly on suitably polished faces (Fig. 1) or after etching a few seconds in boiling HCl. Specimens were found to deform on $\{111\}$ slip planes. An electron microscopic complementary study has shown