Observations of arc-plasma sprayed ferrite films on alumina substrates

Details of the arc plasma spraying process as applied to the production of nickel and nickel-zinc ferrite films have been described in an earlier paper [1]. Further analysis of the films has revealed several important features in the arc-plasma process. Two powders, with the composition $Ni_{0.35}Zn_{0.65}Fe_2O_4$, were used in this investigation, the powders being produced by ball milling and sieving such that they had particle sizes of ~5 μm in the fine powder and 39 to 54 μm in the coarse powder.

Films sprayed onto stainless steel substrates were easily detached after cooling and were used to check the density of the plasma sprayed material by weighing in water. Densities obtained varied between 5.1 g cm^{-3} when sprayed at 400 A to 5.24 g cm^{-3} at 600 A. This compares with a density of 5.4 g cm^{-3} for the starting material.

The surface roughness of the sprayed films was measured using a Rank Instruments Talysurf and typical roughnesses are shown in Table I.

TABLE I Surface roughness of films

Powder	Arc current (A)	Substrate cooling	CLA (μm)	Substrate temperature (°C)
Fine	400	None	2.7	~800
	500		3.6	970
	600		3.8	1150
Coarse	500	None	5.9	970
	600		7.9	1150
Fine	500	Water cooled	2.6	< 200

This shows that coarser powders give rougher surface finishes than fine, and conditions which lead to a higher substrate temperature also lead to increased roughness. The importance of substrate temperature is emphasized by the last entry in Table I, where an improvement in surface finish is shown to be produced by reducing the substrate temperature by means of a water cooled holder. The powders used in this investigation apparently gave a better surface finish than that previously reported [1]. This may be attributable to the very fine powder size (1 to 2 μm) used in the earlier work, resulting in agglomeration prior to plasma spraying.

Surface finishes were also studied by means of the scanning electron microscope, a typical micrograph being shown in Fig. 1. Examination of this photograph, which shows the surface of a film sprayed at 500 A with the fine powder, shows evidence of some surface melting and agglomeration of the individual particles, together with some cracks in the film. These cracks are more clearly seen in polished specimens (Fig. 2) where they are seen to occur as a "crazed" pattern. This cracking is caused by a mismatch in the coefficients of thermal expansion of ferrite (8.5 to 9 × 10^{-6} °C) and alumina

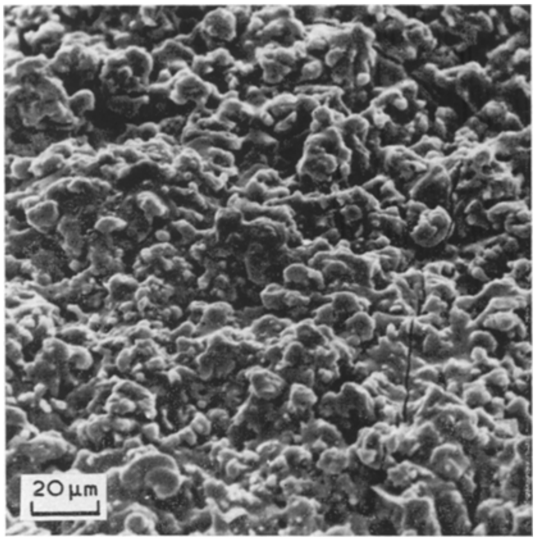

Figure 1 Scanning electron micrograph of a plasma sprayed film showing the typical surface finish.

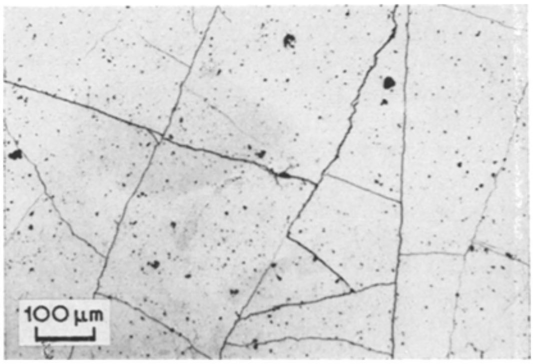

Figure 2 Polished surface of a plasma sprayed ferrite film exhibiting a "crazed" network of cracks resulting from the thermal mismatch between the film and the substrate.

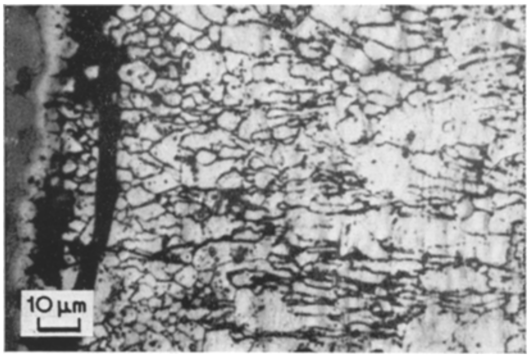

Figure 3 Polished and etched section of a nickel zinc ferrite specimen as-sprayed with fine powder. The cracking along the substrate/film interface results from "pluck-out" during polishing and etching.

(7×10^{-6} °C) such that a tensile stress is introduced into the film on cooling from the spraying temperature.

The microstructure of polished cross-sections was revealed by etching in hot 50% hydrochloric acid. A typical microstructure of a film sprayed with the fine powder at 600 A is shown in Fig. 3. It is immediately obvious that this displays close similarities with chill cast materials in that it consists of a chill layer of fine equiaxial grains close to the substrate together with long columnar grains growing out from this layer; a structure which must result from unidirectional heat flow through the substrate due to the large surface area and small cross-section of the film. Running across the structure are a number of light and dark bands. Estimation of the distance between two consecutive bands suggests that this represents the thickness of material deposited during one pass of the substrate scanning mechanism. The bands represent regions of varying composition, as powder that is injected into the centre of the plasma jet is heated more than the material that only enters the periphery, and thus suffers more decomposition. This has been confirmed by electron microprobe analysis which showed the bands to represent areas of varying zinc content (Fig. 4). The observation that the length of the columnar grains is greater than the thickness deposited in one pass shows that subsequent layers can be deposited epitaxially on top of one another. Such behaviour would suggest that the top layer of the film is close to its melting point.

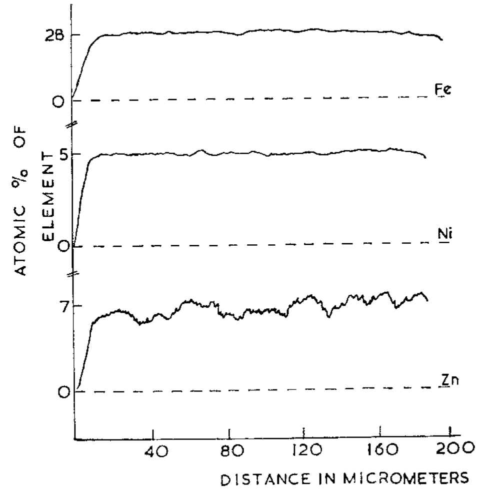

Figure 4 Microprobe analysis scans across a polished cross-section of a film plasma sprayed with the fine powder, showing compositional variations across the thickness of the film.

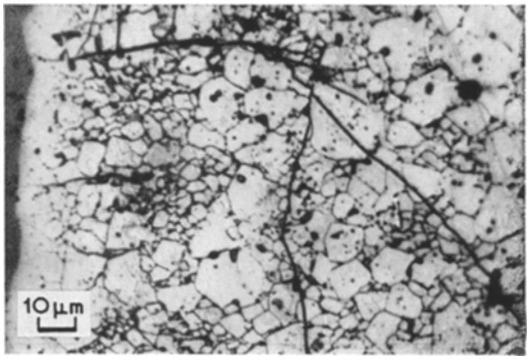

Figure 5 Polished and etched section of Ni-Zn ferrite after annealing at 1200° C.

After annealing at 1200° C, the grain structure is refined and the columnar structure has practically disappeared (Fig. 5). The small grain structure of the chill layer remains and a bond region is visible where interdiffusion between the film and the substrate has occurred.

The microstructure of the films sprayed with the coarse powder are completely different, as shown by Fig. 6. No columnar growth is displayed, and the chill layer is very thin. The majority of the film consists of large grains of a size similar to that of the powder used. This suggests that the larger particles are not melted in the flame, but only softened, being compacted into a film by the force of impact.

The composition of the sprayed film has been

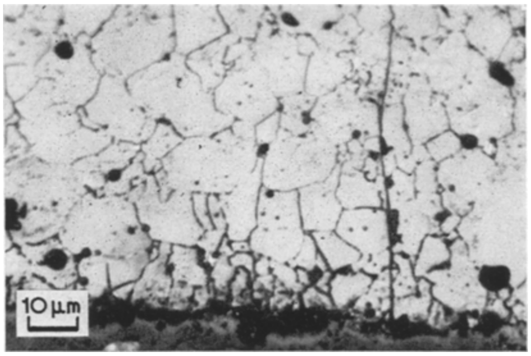

Figure 6 Polished and etched section of Ni-Zn ferrite as-sprayed with the coarse powder.

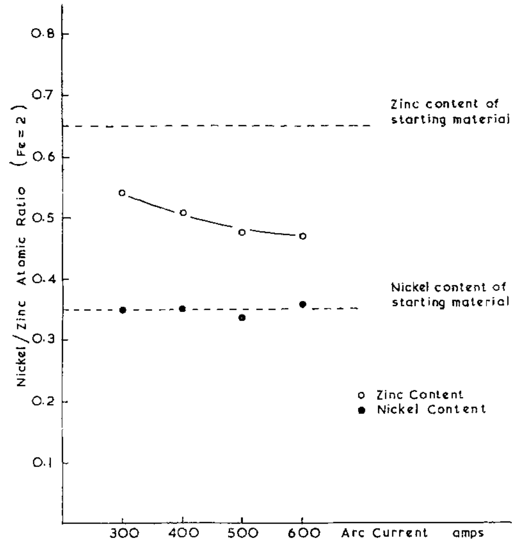

Figure 7 Effect of plasma spraying on the composition of $Ni_{0.35}$ $Zn_{0.65}$ Fe_2O_4 ferrite.

determined using a Pye Unicam 1900 atomic absorption spectrophotometer, the results being shown in Fig. 7. It can be seen that the nickel to iron ratio decreases with increasing arc current. This probably results from an increase in the dwell time of the powder in the flame since the flame is observed to lengthen as the arc current is increased.

The dependence of initial permeability on arc current through its effect on substrate temperature (Fig. 8) clearly shows the importance of arc current as a parameter in the process. Films which have been annealed at 1000° C after spraying all have permeabilities of approximately the same value, irrespective of arc current. The probable effect of increasing the substrate temperature is to cause a decrease in the thermal gradients across the film and thus to reduce the microstresses within the film. Since permeability is structure sensitive, a reduction in stress causes an increase in permeability. Macrostresses caused by the mismatch in thermal expansions of the film and substrate are not affected by either increasing the substrate temperature or by annealing. Oxygen pick-up during annealing or spraying on to hot substrates may affect the results.

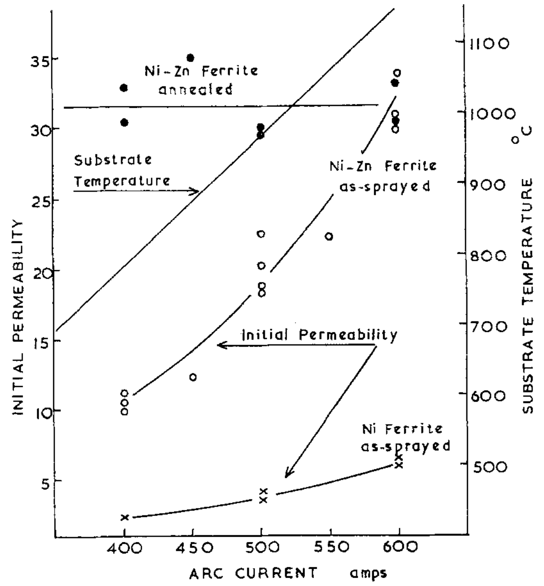

Figure 8 Effect of arc current on substrate temperature and the initial permeability of Ni and Ni-Zn ferrites.

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Reference

1. I. PREECE and C. W. D. ANDREWS, *J. Mater. Sci.* 8 (1973) 964.

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