The electron probe microanalysis of electrical discharge machined die surfaces

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The elemental concentration and distribution characteristics of electrical discharge machined die surfaces have been investigated to a depth of 150 μm using the techniques of electron microprobe analysis. General results indicate a correlation between elemental concentration changes such as Carbon gradiant and Manganese distribution, and other spark eroded surface characteristics previously determined by more conventional metallurgical techniques.

1. Introduction

Electrical discharge machining (EDM), sometimes called just spark-machining or spark-erosion, is a widely used production method utilised by tool manufacturers for the fabrication of die-casting tools, forging dies etc [1, 2]. The principle is well known and involves the production of an electrical discharge in a fluid dielectric located between two electrodes, with the accompanying erosion of one of the electrodes.

By suitable adjustment of the spark frequency and mark-space ratio the cathode can be made to erode and is thus the workpiece whilst the anode is the tool and is generally made of a highly conducting material such as copper or graphite. During the work cycle the tool is gradually fed towards the workpiece and the latter eroded until it has the same surface configuration as the tool.

Whilst possessing definite economic advantages in production, and facilitates the attainment of a given surface finish, it is recognized that for certain applications the high thermal stresses at the surface — which attains localized temperatures of anywhere between 10^4 and $5 \times 10^{4\circ}$ C — give rise to large tensile residual stresses and microcracking and other effects [3, 4] such as element migration which can lower fatigue life. This is particularly true of the die-casting tool.

It was for the latter reasons that the above laboratory undertook on behalf of the Royal Mint an investigation into the surface structural changes undergone by certain die casting tools produced

by EDM, and which had suffered premature and catastrophic failure. The investigation format concerned the constituent elemental concentration and distribution changes within the surface layers and was carried out using the techniques of electron probe microanalysis (EPMA).

## 2. Experimental techniques

2.1. Sample preparation

The samples submitted for analysis were of coining die tools made to BS4659 BO1 material 1% carbon, 1.2% manganese steel. The material had been hardened at 800 to 820° C, oil quenched and tempered at 180 to 200° C. At this stage the hardness figures quoted were 750 to 800 VPN. EDM was carried out using a paraffin dielectric and copper electrodes. The complete specification of the process is shown in Appendix 1. The samples were sectioned and polished perpendicularly to the surface of the die to provide edges on which existed both spark and non-spark eroded areas suitable for examination. It was ensured during sectioning that an area sufficiently far below the eroded edge should be available to provide a "bulk" analysis.

2.2. Analysis procedures

The measurements were carried out using a Scanning Electron Probe X-ray Microanalyser Type SEM-2A (A.E.I. Ltd). Beam voltage used was 30 kV (12 kV for carbon) which was sufficient to excite both K and L spectra up to atomic number

Figure 1 Spectrometer "scan" showing position of Bragg angle peaks for elements present against relative intensity of X-rays.
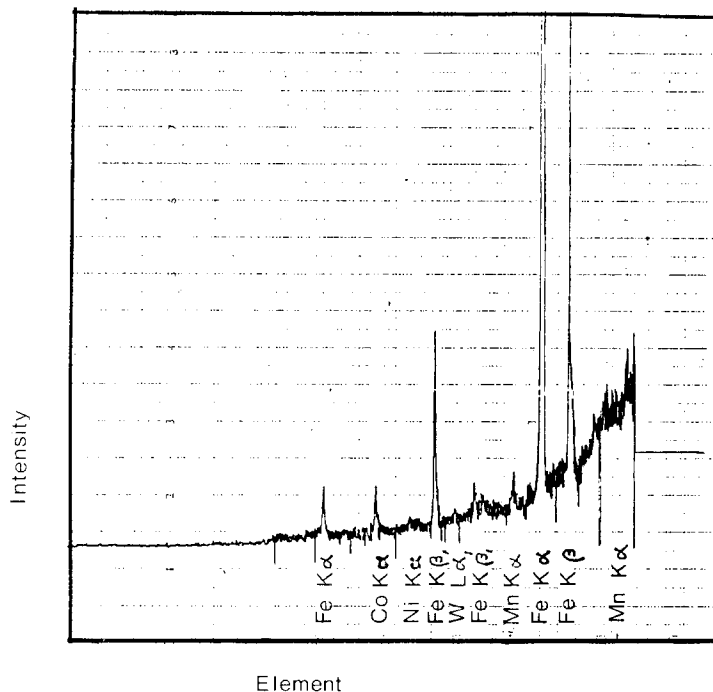
50 and this provided unambiguous identification of any X-ray line. Beam current was of the order of 0.1 μA, and was stabilised using the variations in aperture current in a negative feed-back loop to the condenser lens stabiliser circuit. Stability in this mode is better than 1 in 10^4 over 5 min. Data collection times (total) never exceeded 3 min on each element. Elemental standards were used at all times in rough count assemblage for quantitative analysis.

As a preliminary control experiment, micro-analysis was undertaken on "bulk" samples, at least 10 mm below an edge which had been spark eroded, it being known [5] that structural changes do not occur due to EDM below, at the most, only 10% of the figure shown. At the same time a "scan" through all the elements from atomic number 6 upwards was undertaken to establish the presence of impurities, if any, not expected in the specification. Analysis was then undertaken on the layers immediately underneath (a) the spark eroded (b) the non-spark eroded surfaces of the die sample provided, to a depth of 150 μm in 15 μm steps. The layers were scanned over areas measuring 10 μm $\times$ 30 μm in preference to taking spot counts with a stationary beam, as surface artefacts were often of the same order of size as beam diameter (1 μm). The larger dimension in the above scanned area was parallel to the eroded surface.

3. Results and discussion
3.1. Spectrometer "scan" from atomic number 6 upwards on "bulk" samples
Fig. 1 reproduces typical scans using a mica analysing crystal on bulk samples. It is apparent that elements present in measureable quantity are Fe, Mn, W (and C) with the presence in very small concentrations of Ni and Co. Carbon is analysed using a lead stearate crystal.

3.2. Bulk analysis
Bulk analysis over several areas (250 μm $\times$ 250 μm) of samples EDI–ED3 provided elemental composition and mean concentration tabulated in Table I, shown together with the expected specification.

TABLE I

Element	Concentration	Specification
Fe	97.23%	97.40%
Mn	1.46%	1.20%
W	0.38%	0.50%
C	0.92%	0.90%

The traces of Ni and Co were present in concentrations less than 0.01%. The usual corrections for atomic number (Z) absorption (A) and fluorescence (F) were applied to give the above figures, the figures being the result of 5 successive convergent iterations. ZAF correction procedures were applied to all data contained in this report.

3.3. Microanalysis of non-spark eroded edge

Comparison analyses were performed on non-eroded edges and Fig. 2 shows the elemental concentrations in sample EDI to a depth of 90 μm in 15 μm stages below the surface.

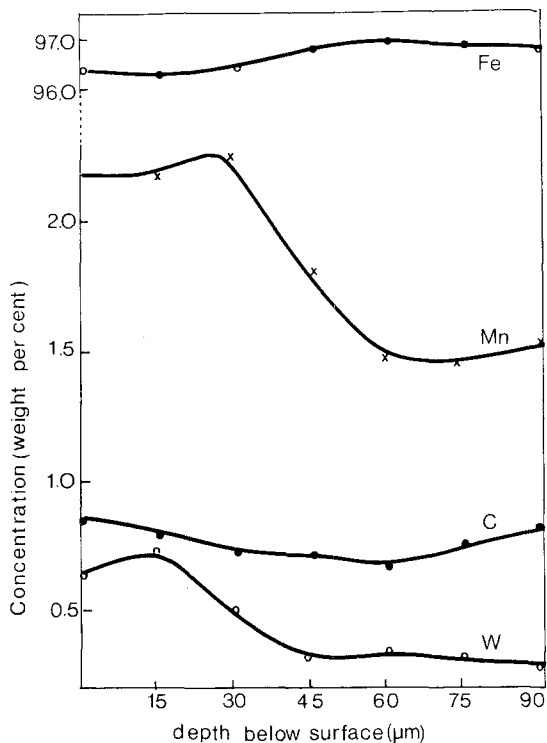In every case the curves are asymptotic to the bulk concentration below 45 μm.

Figure 2 Elemental concentration (in wt %) against depth below uneroded surface (sample EDI).

The previous history of the die material is clearly significant (Fig. 2). Whereas the elemental concentrations might be expected constant in depth in the uneroded specimens, the Mn and W concentrations are elevated to a depth of 45 μm, only thereafter attaining the concentration of the bulk material. Species mobility and consequent overall distributions (as opposed to grain-boundary migration of Mn, for example, which is well known [6]) with variously pre-heated dies is at present being investigated in conjunction with other spark erosion parameters.

3.4. Microanalysis of spark eroded edge

Fig. 3 reproduces the elemental compositions in sample EDI also to a depth of 90 μm in 15 μm stages below the spark eroded surface, and indicates the microscopic trends in concentrations.
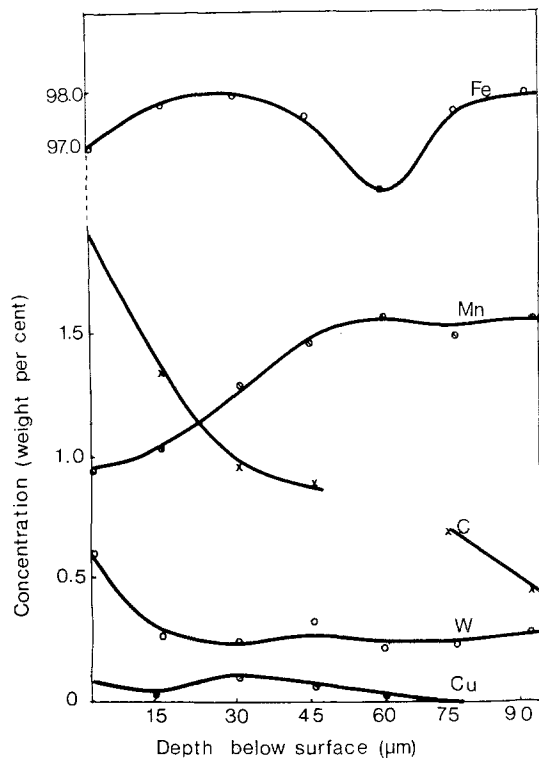

Figure 3 Elemental concentration (in wt %) against depth below eroded surface (sample EDI).

The "gap" in the carbon plot has a significance which is discussed below. It is also significant to note that the copper concentration which appears in Fig. 3 disappears between 60 μm and 75 μm below the eroded surface.

After spark erosion the situation is therefore significantly different. Thus, only at a depth of greater than 90 μm do the elemental concentrations, in every case except carbon, attain that of the bulk material. The carbon gradient itself disappears before 150 μm depth attaining bulk concentration of 0.92% at 145 μm, and agrees with the conclusions of other authors [5, 7] regarding the extent of carbon migration within the range of the EDM process.

The Mn concentration rises from a surface concentration of below 1% to bulk concentration within 60 μm. This is in complete contrast to the uneroded edge.

The carbon concentration at the surface is also very high (1.9%) decreasing rapidly to bulk value as mentioned previously, but having significant characteristics. The point (at 60 μm) which should be in the "gap" is in fact off the top of the low concentration curve and is omitted to avoid confusion. The carbon concentration at this point is

2.8% (averaged over the $30\,\mu m \times 10\,\mu m$ area) and corresponds to the dip in the Fe curve at this point. It represents a large carbon interstitial or inclusion, of which there were found many and could not be avoided.

Since the SEM–2A is fitted with an air-leak carbon decontaminator, which effectively eliminates carbon contamination from the vacuum pump oil, it is possible that the elevated level of carbon encountered is due to the fact that a hydrocarbon dielectric was used in the spark erosion process.

The copper concentration (approx. 0.1% at the surface) generally decreases with depth below the eroded surface and is not measurable below 60 to $75\,\mu m$. This is clearly due to diffusion of tool material as it is not present in uneroded specimens.

4. Conclusions

Electron microprobe analysis has been shown to offer a convincing demonstration of the elemental concentration changes which occur after the EDM of tool die workpieces. The distribution in concentrations corresponds in large measure to the distribution of other parameters such as surface hardness, found using more conventional metallurgical methods [5], although no effect has been made to correlate these effects apart from the more obvious variations in carbon concentration, which gives rise to the hard (600 to 1000 HV) surface layers reported by other authors [2, 5].

Appendix I. Samples and machining

Indonesian 2000 Rupee reverse dies.
Balfour Darwin TOHAISI Type 01. Copper electrodes-diameter   38.22 mm,   0.25 mm   form.
Roughing Channels. Spark CN18A684 Current 4 A.
Pulsator A5B6 Time 45 min. Finish 29 charmille.
Finishing Channels. Microfin channel 6 Channels – 3. Time 10 min.
Microfin – 4, channels – 3 Time 10 min.
Microfin – 2, channels – 3 Time 45 min.
Approximate final finish 20 charmille.

Acknowledgements

References

1. P. L. LIVSHITS, "The electron-erosion machining of metals" (Moscow 1957) DSIR translation Butterworths London.
2. LEIF HASSVIK, CLASS-HENDRIK ENGSTROM, "The electrical discharge machining of tool steel." Uddeholm Alkiebolag pamphlet 1975.
3. I. M. GIBBON, *J. Metals & Materials*, 11 (1969) 308.
4. N. M. BARASH, *Metals Engineering Quarterly* ASM 4 (1965) 110.
5. H. K. LLOYD and R. M. WARREN, *J. Iron and Steel Institute* 203 (March 1965) 238.
6. R. G. WARD, *ibid* 203 (September 1965) 930.
7. I. Z. MOGILEVSKII, *Prob. Eleck. obca. Mat, SSSR* (1960) 86.