Characterisation of TiC layers deposited using an electrical discharge coating process

S J Algodi¹, J W Murray², A T Clare² and P D Brown¹

¹Division of Materials, Mechanics and Structures, and ²Institute of Advanced Manufacturing, Department of Mechanical, Materials and Manufacturing Engineering, Faculty of Engineering, University of Nottingham, University Park, Nottingham, NG7 2RD, UK.

Abstract. Electrical discharge machining (EDM) is a non-conventional, high-accuracy machining process for the manufacture of complex shapes, regardless of hardness of the workpiece. There is interest to develop the EDM technique for coating or surface modification by using a powder metallurgy (PM) tool electrode and/or added powder suspended within the dielectric fluid. We report on the EDM deposition of TiC coatings onto stainless steel, using either Cu or TiC electrodes, with and without Ti powder in the working oil. EDM processed layers exhibited hardness values ~ 3-4 times higher than the substrate, emphasising the ability of EDM to impart improved mechanical performance to the surface of austenitic stainless steel.

1. Introduction

Titanium carbide (TiC) is the most widely used ceramic carbide protective coating owing to its high melting point, hardness, oxidation resistance and chemical stability, along with excellent wear resistance and low coefficient of friction [1]. High quality TiC coatings have been deposited using methods such as CVD, plasma spraying, sputtering and ion plating. An advantage of EDM is that it provides for localised coating or surface modification, without need for complicated equipment. Also it can be applied to complex shapes [2,3]. In this context, there is a need to appraise the process-structure-property interrelationships of EDM since this technique is still at the developmental stage.

2. Experimental

It is not easy to improve the wear resistance of austenitic stainless steel because they cannot be hardened by heat treatment. For this work, commercial 304 stainless steel substrates were used, cut into 20 x 20 x 6 mm sections and polished to a mirror finish. The coating work was performed using a die sinking machine (Mitsubishi EA12V; current 10 A; gap voltage 320 V; negative tool polarity; on-time 8µs; off-time 256 µs; machining time 10 minutes). Two types of tool electrode were compared, i.e. a powder metallurgy (PM) TiC electrode and a conventional solid Cu electrode, with and without Ti powder (45 µm) dispersed into the dielectric fluid (hydrocarbon oil) contained within a stirrer tank (figure 1). The workpiece was fixed to the bottom of the tank, while the tip of the electrode was set to touch the workpiece top surface. The electrode moved towards the workpiece under servo-control and the gap voltage was allowed to rise until it became sufficient to ionise the dielectric, with short duration discharges in the gap between the two electrodes, leading to material replacement or alloying on the localised scale. Metallurgical analyses of the workpiece surfaces and the coatings in cross-section was performed using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffractometry (XRD) and microhardness testing.
3. Results & Discussion

Figures 2(a-d) present secondary electron (SE) micrographs of EDM processed samples, prepared with Cu and TiC electrodes, with and without Ti powder. The surfaces produced using the conventional Cu electrode, without Ti powder, exhibited uniform cracking, voids and ‘volcano-like’ structures (figure 2a), attributable to high thermal energy followed by rapid cooling during discharges. With the addition of Ti powder, near surface porosity and cracking were almost eliminated (figure 2b). Individual and overlapping craters could be distinguished, indicative of less intensive melting, with previous craters left unaffected, noting that the addition of powder to the dielectric fluid resulted in reduced resistance, increased gap distance and consequent reduction in electrical discharge power density [1].
Cracking and porosity were more prevalent on the surface produced using a TiC PM electrode (figure 2c). The porosity may be attributed to the expulsion of gas bubbles from molten material during solidification [4]. It is noted that the melting point of TiC (3143 K) is slightly higher than the boiling point of Fe (3023 K), so when the TiC layer started to solidify, Fe would still be molten, and cracks would tend to develop within the TiC layer upon gradual cool-down due to differential thermal contraction [5]. The addition of Ti powder to the dielectric combined with the use of a TiC electrode led to the production of smoother, denser coatings than those produced with a Cu electrode (figure 2d). In particular, differently shaped dimple features were present which were found to be rich in Ti (figure 2d). This effect was attributed to the movement of Ti powder to form clusters between the two electrodes leading to a localized reduction in the dielectric fluid resistance, promoting discharge, along with the breaking of clusters of TiC powder from the PM electrode which deposited on the workpiece surface, forming dimpled shapes [6]. A ~ 50% reduction in mean crack density was determined for the Cu electrode / Ti powder samples, as compared with samples prepared using the Cu electrode only. Similarly, a ~ 30% reduction in crack density was observed for the TiC electrode / Ti powder samples, as compared with coatings produced using the TiC electrode only.

The crystal structures of the EDM processed samples were appraised using XRD (figure 3). For coatings processed with the Cu electrode, the evidence showed the formation of Fe₃C due to reaction between the substrate and the hydrocarbon of the dielectric fluid (figure 3b), whilst addition of Ti powder led to the formation of TiC and the inhibition of Fe₃C (figure 3c). Surfaces prepared with the TiC electrode exhibited TiC rich phases (figure 3d). EDS analyses were performed to appraise the chemical compositions of the processed coatings in cross-section geometry. Significant amounts of C were associated with surfaces prepared using the Cu electrode, again consistent with the formation of Fe₃C. EDS line profiles suggested that these surface modified layers were ~ 5.5 µm in depth, below which Cu was not detected (figure 4a). The addition of Ti powder led to the development of a uniform
TiC layer ~ 8 μm in thickness (figure 4b) with consequent inhibition of Fe₃C formation in keeping with the XRD data. Much higher levels of Ti were found for EDM samples processed using the TiC electrode (figure 4c) whilst the addition of Ti powder led to the development of a banded profile, indicating initial reaction dominated by the powder in the dielectric fluid, prior to deposition from the TiC electrode (figure 4d, I and II, respectively).

Knoop hardness measurements were performed in cross-section on all samples using 20 indents for each and with a low force of 10 g (figure 5). It was evident that the EDM processed layers exhibited hardness values ~ 3-4 times higher [2,7] than that of the substrate, emphasising the ability of the EDM process to impart improved mechanical performance to the surfaces of austenitic stainless steel. Large variations in indent size were observed for all samples, attributable to local variations in composition and microstructure. The coatings produced using TiC electrodes without Ti powder showed the highest mean values of hardness of ~ 2800 HK (figure 5), consistent with the enhanced XRD TiC profile of figure 3d, as compared to figure 3e, suggesting denser TiC formation. The increasing hardness in the surface modified layers processed with the Cu electrode, with and without Ti powder in the dielectric fluid, was attributed to the development of TiC and Fe₃C, respectively. Further enhancement of hardness in the surface modified layers produced using the TiC electrode was achieved in the presence of Ti powder, but maximised in the absence of Ti powder, again suggesting that the electrode contributed higher quality TiC into the surface modified layer. The EDS line profile transition indicated in Figure 4d suggested local depletion of Ti powder, with TiC from the electrode dominating the latter stages of deposition.

4. Summary
The novel EDM technique as used to deposit TiC coatings on stainless steel substrates has been appraised. Structure determination confirmed the presence of TiC within the recast layer, deposited using a TiC electrode. The addition of Ti powder to the dielectric oil had significant impact on the coating process with the production of smoother surfaces free of cracking and porosity. However, coatings with highest levels of hardness were produced in the absence of Ti powder.

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