



Full Length Article

Investigation on the surface layer formed during electrochemical modification of pure iron

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ABSTRACT

This paper presents an experimental and analytical investigation on electrochemical modification of pure iron, aiming to obtain a surface layer which has good machinability for further processing. The microstructures of the modified layer were compared from a relative low current density (10 A/cm²) to high current density (30 A/cm²) and characterized by various detection equipment. A detailed X-ray photoelectron spectroscopy (XPS) analysis was conducted to measure the composition profile of the black film on the modified layer. The results show that, during electrochemical modification, the subsurface formed a loose porous layer covered with a flimsy oxide. At low current density, uniform dissolution was obtained and the surface was not corroded off completely. However, at large current density, due to the strong electrochemical action and uneven flow field, the cross section of pure iron became waved and the surface shape deteriorated. The results of the water contact angle measurements show that the surface hydrophilicity was enhanced by electrochemical modification, which might improve the cooling effect of the machining region. The XPS analysis indicated a bi-layer structure of oxidation film on the modified layer and the outmost Fe³⁺ species layer was considered to be the oxidation of Fe²⁺ species from inner layer.

1. Introduction

Pure iron, with its high saturation magnetic induction, high magnetic permeability and low coercivity, is widely used in electron, military industry and other fields [1,2]. However, as with other ferrous metals, ultraprecision machining of pure iron faces the issues of serious tool wear, which caused by the graphitization of diamond [3]. Besides, high plastic deformation of pure iron chip easily adheres on the cutting tool and causes serious work hardening that makes it extremely difficult to cut and also hard to guarantee the machining surface quality [4]. Moreover, due to the low stiffness and large deformation [5], thin-walled weak-rigidity structural parts of pure iron, such as shaped charge liner, are extremely sensitive to the cutting force and thermal load. Therefore, weakening or suppressing the negative effects of cutting force, thermal load during processing, and developing new material removal methods are crucial ways to solve the problem of extreme manufacturing of weak-rigidity components.

Among various non-traditional machining techniques, electrochemical machining (ECM) could be a good solution for its almost stress free, no thermally influenced machining zones [6]. According to the principle of the ECM technology, the material is removed from the workpiece by anodic dissolution in aqueous electrolytes at very large

current density and high flow rate [6–8]. The possible reactions of Fe during ECM are listed in Eqs. (1)–(3) [6]:



However, the evolution of oxygen in parallel makes the situation more complex, as shown in Eqs. (4).



The presence of ferric ions and oxygen indicate the formation oxide layer [9], such as FeO, Fe₂O₃, Fe₃O₄ et al. In the condition of paper [10], only oxygen was generated at current densities < 5 A/cm².

In the past decades, the attention of ECM of pure iron was mainly focused on large current density and high flow rate. An electrochemical device, the scanning droplet cell, was designed for electrochemical surface analysis and then improved by establishing a capillary-based droplet cell to simplify the simultaneous handling of two capillaries [11,12]. With the designed setup, Rosenkranz [9] studied the surface structure during pulsed ECM of iron in NaNO₃ at large current density

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($i = 33 \text{ A/cm}^2$) and high electrolyte speed ($v_{el} = 20 \text{ m/s}$). The different reaction products and mean current efficiency were analyzed quantitatively by microcell UV–Vis spectrometer setup, and a time resolved model was extended for the surface structure during ECM. Lohrengel [6] gave a tentative scheme of duplex model to describe interface structure: a modified solid passive film and a viscous adherent layer of supersaturated iron nitrates. With respect to the composition of oxide film, Rosenkranz [9] measured the products in the electrolyte and indicated that the composition was strongly dependent on the pulse length.

Among the aforementioned studies, the current densities and flow rates adopted during ECM are generally very high, because the large current density and high flow rate could enhance the machining efficiency and improve the surface roughness. Nevertheless, it is very difficult for ECM to process components with high machining accuracy, such as precise plate with high flatness, spherical shell with high dimensional precision. As an alternative way, ECM at relatively low current density and flow rate can be utilized to obtain a modified layer with better machinability, because it can significantly change the microstructure. Combined with mechanical method, low stress processing and extended tool life might be obtained.

Furthermore, it is well known that the diamond tool suffers significant wear when cutting iron material due to the graphitization. As the tool is mostly in direct contact with the black film covered on the layer, thus, the modified surface layer may suppress the wear of diamond tool and the tool wear mechanism would change. Therefore, it is extremely essential to investigate the composition of the black film covered on the modified layer for further studying the mechanism of the diamond tool wear.

In this paper, electrochemical modification for pure iron surface was investigated to improve the machinability. The modified layers were characterized and compared from a low current density (10 A/cm^2) to a relatively large current density (30 A/cm^2) by various detecting methods. In addition, the wetting properties of the machined surfaces were measured. In order to get a deep view of the black film on the surface, a detailed study on the compositional depth profile of the film was conducted by X-ray photoelectron spectroscopy (XPS) analysis with Ar^+ ion sputtering for its widely used in characterizing the thin layers [13–18].

2. Experimental

2.1. Workpiece preparation

Pure iron (DT4E) was used as the workpiece which was a cylinder with a diameter of 15 mm and a height of 5 mm. The workpiece was polished by SiC paper of P600, P800, P1000, and P2000 sequentially, put in acetone solution to remove oil, dried and clamped to the electrochemical modification system, as shown in Fig. 1. The experimental parameters were illustrated in Table 1. After modification, the treated surface of the workpiece was covered with epoxy resin and the cross section was polished by cross-section angle polishing method, as shown in Fig. 2. With this method, the thickness (H) was calculated by the following formula, when $\theta = 5.739^\circ$, the thickness (H) was magnified by ten times and became more intuitive to observe. Furthermore, the grains observed at the interface could approximately reflect the shapes and sizes of the protrusions at different heights, as shown by the green line in Fig. 2.

$$H = L \times \sin \theta \quad (5)$$

2.2. XPS

The surface film was chemically characterized by XPS which was performed in UHV chamber by using a monochromatic Al $K\alpha$ X-ray source operated at 12 kV and 8 mA. For detailed in-depth information

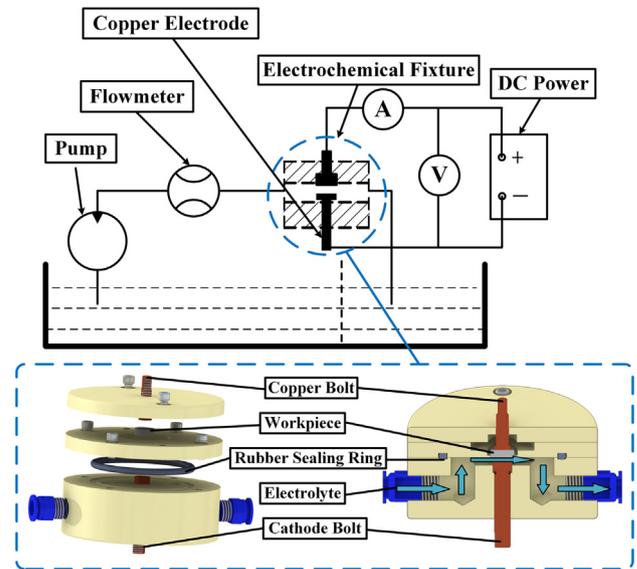


Fig. 1. Schematic of the electrochemical modification system.

Table 1

Parameters of electrochemical modification.

Processing parameter	Value
Current density (A/cm^2)	10, 20, 30
Frequency (kHz)	10
Duty ratio	0.5
Electrolyte	NaNO_3 (10 wt%)
Inter-electrode gap (mm)	0.9
Process time (s)	60
Electrolyte flowrate (ml/s)	100

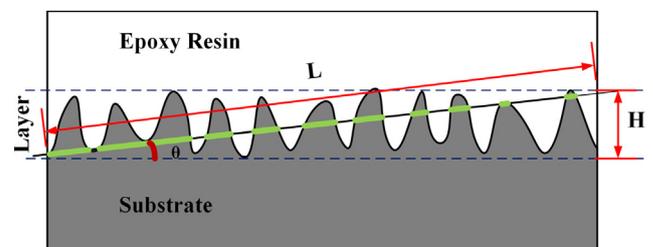


Fig. 2. Illustration of the cross-section angle polishing method.

of the surface film, in this paper 20 eV pass energy with a step size of 0.1 eV was employed for high resolution measurements and the sputtering time ranged from 15 s to 2025 s by 2 keV Ar^+ ions sputtering. Data processing was carried out by using CasaXPS software, all the spectra were corrected by applying adventitious C 1s signal at 285 eV. A non-linear Shirley backgrounds were subtracted and Gaussian-Lorentzian peak shapes were applied for curve fitting. Due to the spin-orbit coupling, the peak ratio of Fe $2p_{3/2}$ and Fe $2p_{1/2}$ was limited to 2:1. In order to obtain good fitting, asymmetric tail functions were used for Fe 2p region during curve fitting. It should be noted that the full width at half maximum (FWHM) in this paper is not constant but varies in a certain law because Fe^{2+} or Fe^{3+} FWHM increases with Fe^{2+} or Fe^{3+} content [15].

3. Results and discussion

3.1. Morphology

The digital images of the machined workpieces are shown in Fig. 3(a–c). As can be seen, the smooth surfaces became dark and

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