

Surface Modification of Ti-6Al-4V by Gas-liquid Mixed EDM

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Abstract

Ti-6Al-4V alloy is widely used in many fields due to its excellent properties. However, its further applications is limited by its low hardness and poor wear resistance. In this paper, gas-liquid mixed electrical discharge machining (EDM) process was applied for surface modification of Ti-6Al-4V alloy. The multi-hole electrode was adopted to flow nitrogen to mix nitrogen with special spark oil. The effect of different parameters (peak current and pulse duration) on surface morphology, cross section morphology, micro hardness and wear resistance were investigated. The results indicated that the gas-liquid mixed EDM process has better performance on sample surface with fewer pores and shallow craters. A continuous and thick recast layer was obtained by gas-liquid mixed EDM process and N element was migrated to the sample surface from nitrogen gas. The XRD results demonstrated that TiN hard phase was formed on the sample surface, thus the micro hardness was nearly three times higher than that of the matrix, reaching 1329.5HV, and the wear resistance is improved accordingly. The surface of Ti-6Al-4V alloy was modified by gas-liquid mixed EDM process.

1 Introduction

Ti-6Al-4V alloy has extensively used in aerospace, automotive and biomedical applications owing to its prominent characteristics, including high specific strength and excellent corrosion resistance [1–3]. However, titanium alloy has low hardness and poor wear resistance, which restrict its further application especially in wear and tear engineering [4–5]. Hence, it is essential to find an effect approach to enhance the mechanical performance of titanium alloy. Titanium alloy is also difficult to process with traditional method [6]. Electrical discharge machining (EDM) is a non-traditional machining method with no direct contact from the workpiece material and the tool electrode, it makes use of a series of spark energy between electrodes to melt and evaporate materials irrespective of their mechanical properties [7, 8]. However, the EDMed surface of titanium alloy still has poor surface quality like micro cracks and pores, low wear resistance.

To further improve the surface properties of titanium alloy, researchers have attempted many ways to achieve surface modification. Yan et al. [9] studied that adding urea into the distilled water to machine pure titanium, their results indicated that TiN was formed on the machined surface due to the reaction between the workpiece and urea solution, thus the wear resistance and hardness was improved. Unses et al. [10] improved the machining performance of titanium alloy with graphite powder mixed EDM. Li et al. [11] investigated the surface characteristics of Ti-6Al-4V alloy by adding SiC particles in EDM process with magnetic stirring. It was found that the machined surface was smoother than EDM process, the micro hardness was improved more than two times than bulk material due to TiC and TiSi₂ were formed on machined surface. Öpöz et al. [12] also used SiC powder to process titanium alloy, the results showed that SiC particle was transformed from the dielectric to machined surface which formed a hard layer. Shabgard et al. [13] studied the influence of carbon nanotubes into dielectric to machine Ti-6Al-4V alloy. The results revealed that the suspended carbon nanotubes in dielectric demonstrated better surface quality with less micro cracks and decreased MRR and TWR. Bui et al. [14] discovered the effect of

adding silver nano-particles into the dielectric. The results showed that silver particles were deposited on the workpiece surface, the antibacterial property was also improved due to an antibacterial layer was formed on the medical titanium alloy surface. Sharma et al. [15] improved the surface properties of titanium alloy using micro electric discharge coating. They used hexagonal boron nitride powder with average size 70 nm mixed deionized water. It is concluded that the micro hardness and corrosion resistance were both increased due to the existence of TiN and TiAlN. Similarly, tungsten disulphide powder was added on titanium alloy by Mohanty et al. [16] through micro electrical discharge machining. The machined surface formed a hard and solid-lubricating layer, thus the micro hardness was increased by three times and wear rate was decreased as compared to that of base material. Takezawa et al. [17] surveyed the effect of mixing micro bubbles with the dielectric fluid to machine titanium alloy and steel under the finishing condition. Their results revealed that the formation of nitride in the steel did not improve the hardness, and for titanium alloy material, the nitrogen micro bubble was not necessary for the generation of nitride. Kong et al. [18] supplied submersed gas-flushing EDM method to machine Ti-6Al-4V alloy, the resulted showed the argon medium provides better surface integrity and higher machining efficiency as compared to the air medium, the relative electrode wear ratio was decreased in the argon medium.

From the mentioned literatures above, it was found that so many efforts have been made to achieve surface modification of titanium alloy by the addition of different powders or other substance. However, there are few researches on gas-liquid mixed EDM process to machine titanium alloy, especially for gas-liquid mixed EDM with nitrogen as medium. Nitrogen is being utilized not only as medium, but also as reaction gas at high temperature. The gas-liquid mixed EDM process combines the advantage of gas and liquid processing, the discharge process is more stable when nitrogen is mixed with the special spark oil.

In this paper, the machining properties of Ti-6Al-4V alloy by gas-liquid mixed EDM with different parameters were investigated. The effect of gas-liquid mixed dielectric on the machined performance of titanium alloy was studied by injecting nitrogen into multi-hole electrode, and compared with that of pure liquid dielectric. It was expected that the nitrogen could react with titanium alloy to form TiN compound at the elevated temperature and high pressure of EDM discharge, thus gas-liquid mixed EDM process can improve the machining performance of Ti-6Al-4V alloy.

2 Materials And Methods

Ti-6Al-4V alloy was chosen as the workpiece material in the size of 10mm 10mm 5 mm, and Table 1 displays its chemical composition. The customized multi-hole copper with the size of $\Phi 16\text{mm}$ 65mm was used for tool electrode material, as shown in Fig. 1. The multi-hole electrode can provide a flow of nitrogen as compared with the cylindrical electrode. The nineteen uniform holes with a diameter of 0.8 mm were selected to ensure uniform flow of nitrogen through the tool electrode. The screw thread on the electrode was designed to connect the electrode holder, and the outer diameter of electrode matched the internal diameter of the electrode holder, which can fix the multi-hole electrode. Based on many experiments, the pressure of nitrogen was chosen as 0.02MPa. The reason is that too high pressure

would lead to discharge in gas, or too small pressure would cause complete discharge in liquid. The pressure of nitrogen was adjusted to form a gas-liquid mixed dielectric condition.

The experiment was conducted with die-sinking EDM machine (CNC-A30, Rixin Co., Ltd., Dongguan, China). Fig. 2 (a) shows the experimental schematic, and Fig. 2 (b) displays the photograph of experimental set-up. The multi-hole tool electrode was mounted on the processing head through the electrode holder, one side of the electrode holder is also the inlet of nitrogen, nitrogen as the gas dielectric flows into the machining area by the multi-hole from the electrode. The liquid dielectric is a kind of special spark oil with good electrical conductivity. The nitrogen and special spark oil are mixed in the working slot as gas-liquid dielectric. The properties of nitrogen and special spark oil are shown in Table 2. The sealing ring is connected with the gas supply system to ensure gas tightness during the machining. In order to make nitrogen distributed evenly on the workpiece, spark position shaking on EDM machine is adopted.

The machining properties of Ti-6Al-4V alloy in three different sets of parameters were investigated. The processing variable parameters are shown in Table 3. For all experiments, the other parameters remain unchanged with the pulse off time of 20 μ s and the supply voltage of 60 V, the polarity is positive, and the experiments are performed for 10 min.

After experimentation, the specimen were ultrasonically cleaned by ultrasonic cleaning machine for 10 min, followed by air drying, then using a scanning electron microscope (SEM; Apreo, FEI Ltd., Hillsboro, OR, USA) to observe surface morphology and using energy dispersive spectroscopy (EDS; Apreo, FEI Ltd., Hillsboro, OR, USA) to analyze elemental composition. To observe the cross section morphology, the samples were mounted, polished, ultrasonically cleaned and etched by HF: HNO₃: deionized water in the volume ratio of 1: 2: 7. The micro hardness of cross section was evaluated by the hardness device (FM800, Future tech Cop., Shanghai, China), with a constant load of 50gf and dwell time of 15s. The X-ray diffraction (XRD; D8 Venture, Bruker Ltd., Madison, WI, USA) was adopted to analyze the phase of the machined surface. The wear resistance was performed by the wear tester (MMU-10G, Shijin Group Co., Ltd., Jinan, China), then the worn surface morphology was observed by SEM.

3 Discussion

3.1 Surface morphology

Fig. 3 displays the surface morphology of Ti-6Al-4V alloy at different parameters. Fig. 3 (a) and (b) show the surface morphology of EDM and gas-liquid mixed EDM at small parameters, respectively. It is clear that the EDMed surface has much pores than the gas-liquid mixed EDMed surface, Fig. 3 (e) and (f) show the surface morphology in EDM and gas-liquid mixed EDM process at high discharge parameters, respectively. It can also be seen that the gas-liquid mixed EDMed surface has less pores than the EDMed surface. This may be due to the flow of nitrogen facilitates the molten materials uniformly distributed on

the surface, the pores would be covered by the molten materials, thus the number of pores on the gas-liquid mixed dielectric is reduced.

It can be observed on increasing the processing parameters, the EDMed surface becomes worse. This may be due to the single pulse energy is large at high parameters, and more molten materials are ejected from the molten pool, resulting in the deepening of discharge pit and the deterioration of surface quality [19]. However, it can be seen that with the increase of peak current and pulse duration, the gas-liquid mixed EDMed surface has better surface morphology, which is very different from the EDMed surface. This may be due to that the flow of nitrogen disperses the discharge energy, and the energy per unit area is reduced, which leads to less molten materials are thrown from the molten pool, thus the surface morphology is better than the EDMed surface. The results show that the influence of gas-liquid medium is greater than that of process parameters.

Comparing the gas-liquid mixed EDMed surface (Fig. 3 (b) (d) (f)) and the EDMed surface (Fig. 3 (a) (c) (e)), it can be seen that the gas-liquid mixed EDMed surface is smoother than the EDMed surface at the same parameters. This is due to the addition of nitrogen increases the discharge gap and reduces the energy density, so that the spark energy is uniformly distributed on the machined surface [20]. Besides, nitrogen moves randomly in the discharge channel under the flow of spark oil medium, which leads to the distribution of molten materials more even and facilitates the current dispersion in the discharge process, refines the discharge energy of single discharge and thus makes the machined surface smoother. It is also noticed that the size of discharge crater on the gas-liquid mixed EDMed surface is larger as compared with that on the EDMed surface under the same parameter. This may be due to nitrogen is involved in the spark discharge, and the liquid dielectric mixed with nitrogen has a weaker compression effect than the liquid dielectric [21], thus the discharge channel diameter becomes larger than that of liquid dielectric, which decreasing the energy density. Therefore, the discharge craters are shallower with large diameter. Besides there is additional heat except for the heat generated from the discharge energy. The extra heat may come from exothermic reaction between nitrogen and molten titanium alloy at high temperature, which also lead to larger craters. The similar explanation was also put forward by Singh et al. [22].

3.2 Cross section morphology

Fig. 4 illustrates the cross section morphology of machined surface at different parameters. Fig. 4 (a) and (b) show the cross section morphology of EDMed and gas-liquid mixed EDMed at small parameters. The results show that some cracks and pores could be observed on the EDMed surface, but not on the gas-liquid EDMed surface, and the recast layer of gas-liquid mixed EDM is more continuous than that of EDM. The reason is that nitrogen enters the discharge channel, which disperse the discharge energy and generates a larger range of spark discharge. Therefore, the discharge point is more uniform, reducing the uneven discharge point in liquid EDM. Accordingly, the recast layer of gas-liquid mixed EDM is more continuous and consistent.

With the increase of machining parameters, the thickness of the recast layer increases regardless of whether the dielectric is liquid (Fig. 4 (c)) or gas-liquid mixture (Fig. 4 (d)). That maybe due to the discharge energy enhances with the peak current and pulse duration increase, which leads to more materials melting. However, the proportion of molten material that can be flushed away by the dielectric is constant [23]. Thus, more molten materials re-solidified on the workpiece surface compared with that of small parameters. Interestingly, no matter the parameters are large or small, the thickness of the gas-liquid mixture is thicker than that of the liquid dielectric, which is contrary to the phenomenon studied by Wang et al. [24]. That may be due to the cooling effect on the multi-hole electrode by nitrogen improves the thermal conductivity of the cooper electrode, so that more heat is transferred into the electrode, and relatively little heat is delivered to the workpiece. Although the heat on the workpiece can melt the workpiece, there is not enough heat to throw the molten material out, thus the thickness is thicker. In addition, the heat convection of nitrogen takes some of the heat away and has a cooling effect on the solidification process [25]. Hence, the cooling rate of gas-liquid mixed dielectric is faster than that of the pure liquid dielectric, and some molten materials re-solidified on the surface before being thrown out. Therefore, the recast layer is thicker than that machined in liquid dielectric.

Observing the cross section morphology of the sample obtained in the gas-liquid mixed dielectric (Fig. 4 (b) and (d)), it is noticed that the bond between recast layer and the matrix is dense. Besides, the microstructure of the recast layer is mainly granular and dendritic, moreover, the particles gradually become fine and dense from the matrix to the machined surface, and transit from dendritic to granular. The similar results have been found by Morton et al. [26]. Different solidification rates at different depth lead to different microstructure. During the gas-liquid mixed EDM process, nitrogen is blown into the machined surface, and the machined surface cools rapidly, forming a dense recast layer. However, with the increase of the depth, the internal temperature of the workpiece is higher, the temperature difference increases, thus the cooling rate is slower than the machined surface. Due to the difference of thermal expansion coefficient of titanium alloy, the volume of the internal workpiece is contracted, hence the molecular density changes, forming the dendritic structure.

3.3 Surface element analysis

Different microstructures are usually related to performance. To further explore the cause of different microstructure, two typical samples with different microstructures were chosen for surface element analysis. Fig. 5 shows the surface element analysis of EDM and gas-liquid mixed EDM process at the same parameter, respectively. The analysis region is shown in the red box. Different surface morphology has distinct element content. It can be observed the peak of N element is visible on the gas-liquid mixed EDMed surface, but not on the EDMed surface. The presence of N element on the gas-liquid mixed machined surface confirmed that the migration of N element from nitrogen to the machined surface. That is because nitrogen ionizes under high temperature and high pressure produced by spark discharge, resulting in the deposition of ionized nitrogen on titanium alloy surface. Therefore, the existence of N can

be obtained on the gas-liquid mixed EDMed surface, which lead a different microstructure on the machined surface.

3.3 XRD analysis

To further explore the phase composition of the machined surface, the gas-liquid EDM surface obtained under large parameters was analyzed by XRD, as shown in Fig. 6. Results show that TiN, Ti and Al phase are present on the workpiece surface. The formation of TiN hard phase is owing to the instantaneous energy of spark discharge melts the titanium alloy and forms a molten pool, at the same time, nitrogen decomposes into nitrogen atoms at high temperature, and even ionizes into nitrogen ions. The molten titanium reacts with nitrogen to form TiN hard phase. The formation of TiN proves that the element N in section 3.2 exists in the form of compound. The Ti and Al phases come from the remaining molten titanium after nitriding reaction.

3.4 Micro Hardness

The micro hardness of the recast layer plays an important part in wear resistance. Fig. 7 depicts the distribution curve of micro hardness on the cross section of surfaces machined by EDM and gas-liquid mixed EDM process at high parameter, respectively. Results shows that the micro hardness of gas-liquid mixed EDM process varies between 1329.5 HV and 314 HV, while that of EDM ranges between 801.6 HV and 314 HV. This shows that the micro hardness of gas-liquid mixed EDM is almost 65.9% higher than that of EDM, which is more than three times of that of the matrix material. The improvement of micro hardness is due to the formation of TiN hard phase on the sample surface. Because the research shows that TiN ceramic layer has high hardness [27, 28]. The micro hardness of the machined surface is the highest (1329.5 HV). With the increase of the depth, the micro hardness decreases gradually until it reaches the matrix hardness (315 HV). The micro hardness of the location below the surface 20 μm is 1001.9 HV, and the micro hardness at 60 μm away from the surface is still higher than 679HV, while at the same distance the micro hardness of EDM process is 314 HV, which is the same as the matrix hardness. This variation of micro hardness corresponds to the analysis of cross section morphology as mentioned in Section 3.2 that the thickness of TiN recast layer of gas-liquid EDM is thicker.

3.5 Wear resistance

Fig. 8 illustrates the surface morphology of high parameter machined surfaces obtained by gas-liquid mixed EDM and EDM after wear test. The wear test shows that there are obvious marks and grooves on the EDMed surface, while the marks on the surface of gas-liquid EDM are not obvious under the same parameters, even if the surface features are resemble to the original features of EDM. Thus, the wear resistance of gas-liquid mixed EDMed surface is better than the EDMed surface. That is due to the

formation of TiN hard layer on the sample surface, which has better wear resistance [29, 30]. This is consistent with the improvement of micro hardness discussed in Section 3.4.

4 Conclusions

In this paper, the machining properties of Ti-6Al-4V ally by gas-liquid mixed EDM process were investigated. The main conclusions can be drawn:

1. The gas-liquid mixed EDM process can obtain smoother surface morphology with less pores as compared with EDM process.
2. The recast layer of gas-liquid mixed EDM is more continuous and thicker than that of EDMed surface and its microstructure is mainly granular and dendritic.
3. X-ray diffraction analysis reveals that the hard phase of TiN is formed on the gas-liquid mixed EDMed surface. The average surface micro hardness increases from 315 HV to 1329.5 HV.
4. The micro hardness of gas-liquid mixed EDMed surface is improved by almost 65.9% as compared to that of EDM. With the increase of depth from the surface, the micro hardness decreases gradually until it reaches the matrix hardness.
5. Wear test shows that the gas-liquid mixed EDM process enhances the wear resistance of titanium alloy and realizes the surface modification of titanium alloy.

Declarations

Author contributions Wei Zhang: experiments, writing the original draft, result analysis. Li Li: resources, editing, data curation, project administration. Ning Wang: methodology, investigation, experiments. Jianbing Meng: resources, supervision. Jianhua Ren: conceptualization, resources.

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Compliance with Ethical Standards

Conflicts of interest The authors declare that they no conflicts of interest.

Ethical approval This work has not been published elsewhere.

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Tables

Table 1 Chemical component of Ti-6Al-4V

Chemical composition	Al	Si	K	Fe	V	Ni	Ti
Content/%	5.8	0.152	0.0196	0.2	4.27	0.0304	Rest.

Table 2 The properties of different dielectric

Property	Dielectric strength (MV/m)	Dielectric constant	Dynamic viscosity (g/m·s)	Thermal conductivity (W/m·K)	Heat capacity (J/g·K)
Nitrogen	2.8	1.0	0.01	0.025	1.04
Special spark oil	1.7	1.8	1.64	0.149	2.16

Table 3 Parameter settings

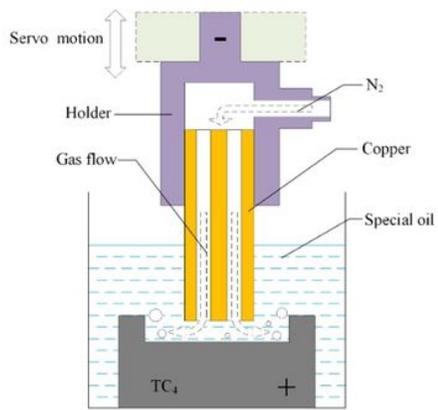
Parameters	Peak current/A	Pulse duration/ μ s
Low parameters	1	18
Medium parameters	3	30
High parameters	5	60

Figures

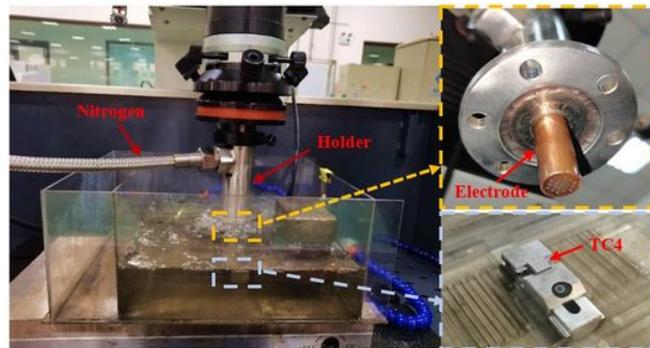


Figure 1

Multi-hole electrode



(a) Experimental schematic



(b) Experimental photograph

Figure 2

Experimental set-up

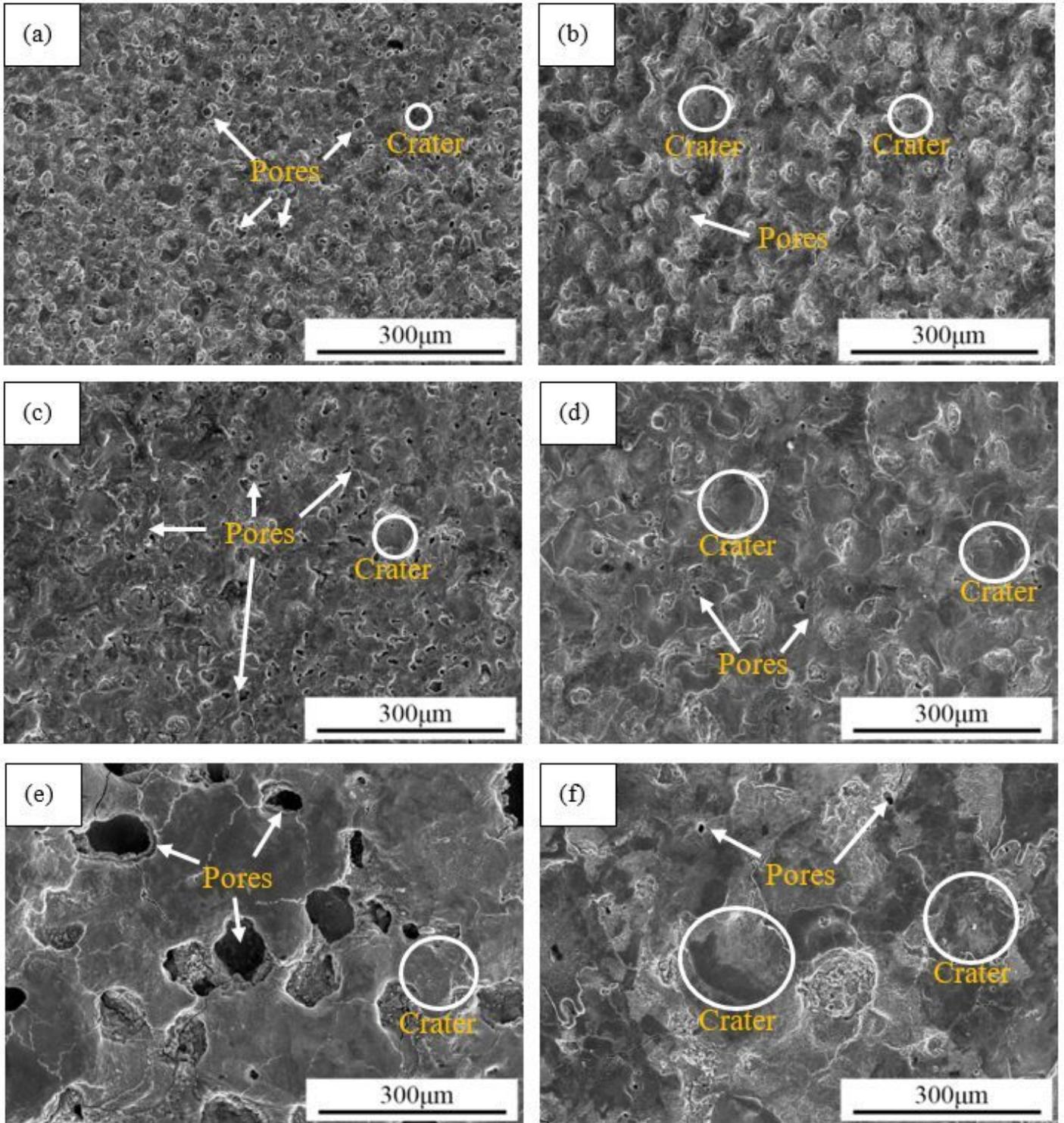


Figure 3

Surface morphology of different parameters (a) Small parameter, EDM; (b) Low parameter, Gas-Liquid mixed EDM; (c) Medium parameter, EDM; (d) Medium parameters, Gas-Liquid mixed EDM; (e) High parameters, EDM; (f) High parameters, Gas-Liquid mixed EDM

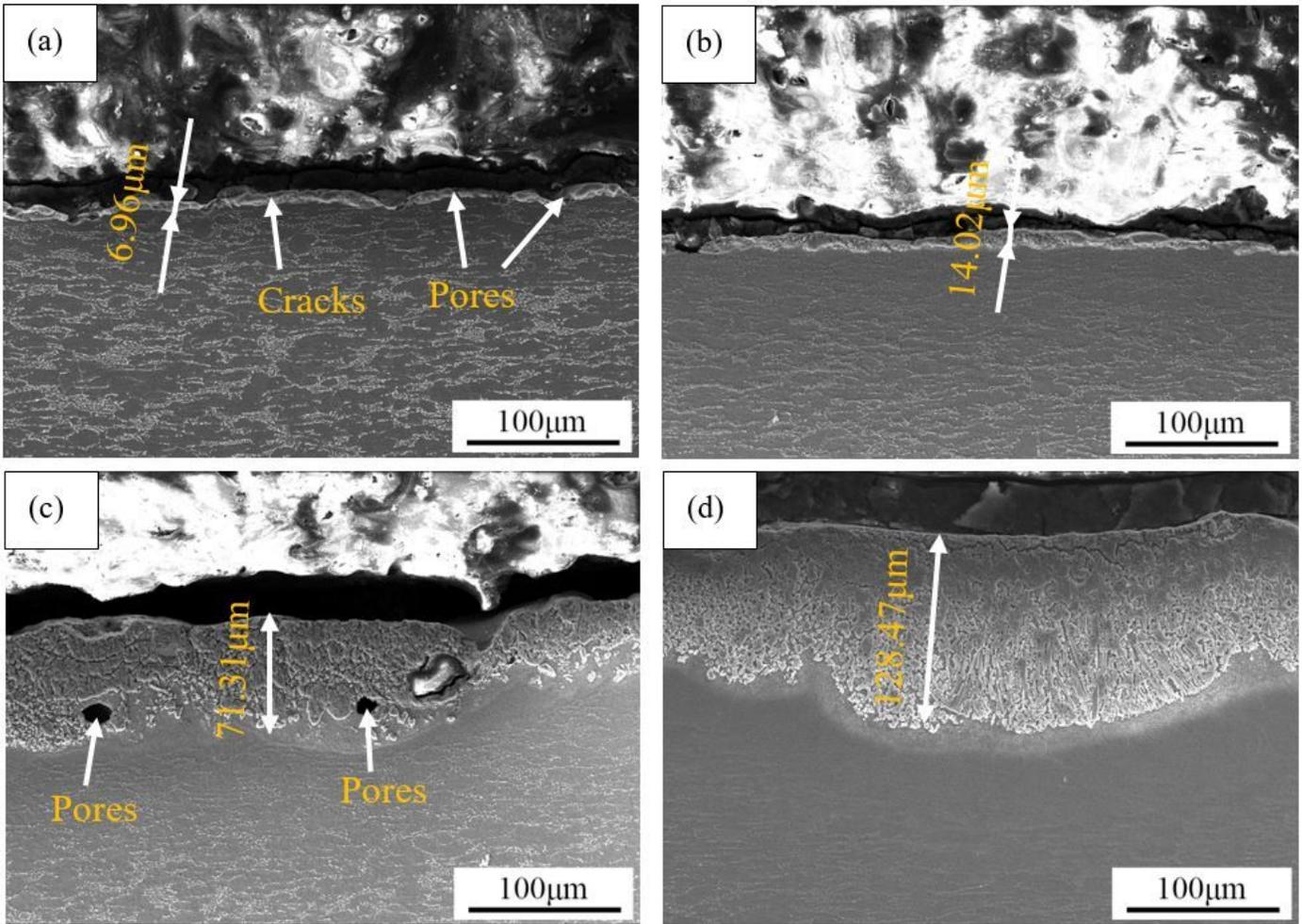


Figure 4

Cross section morphology of different parameters (a) Small parameter, EDM (b) Small parameter, Gas-Liquid mixed EDM; (c) High parameter, EDM; (d) High parameter, Gas-Liquid mixed EDM

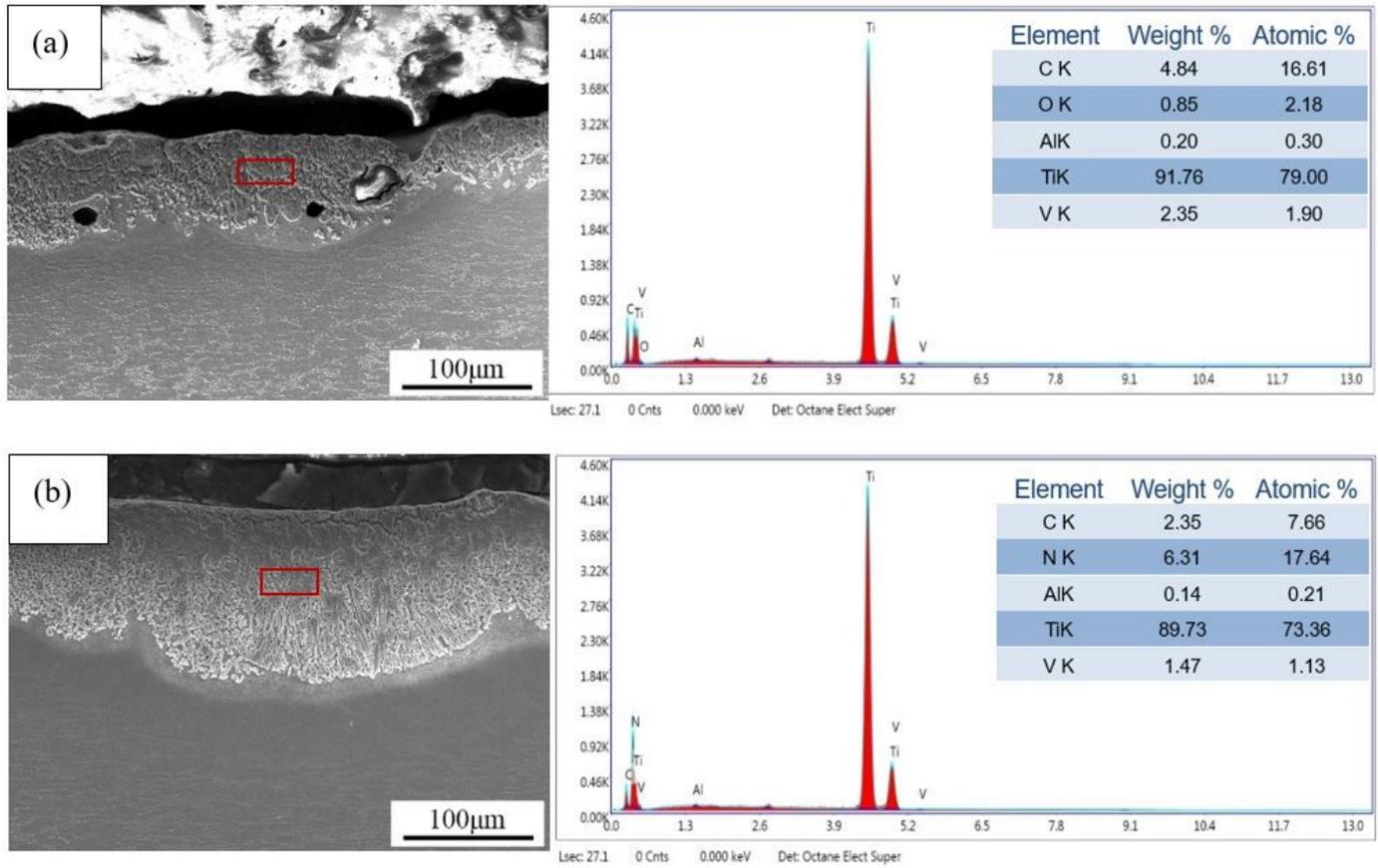


Figure 5

Surface element analysis at high parameter (a) EDM; (b) Gas-liquid mixed EDM

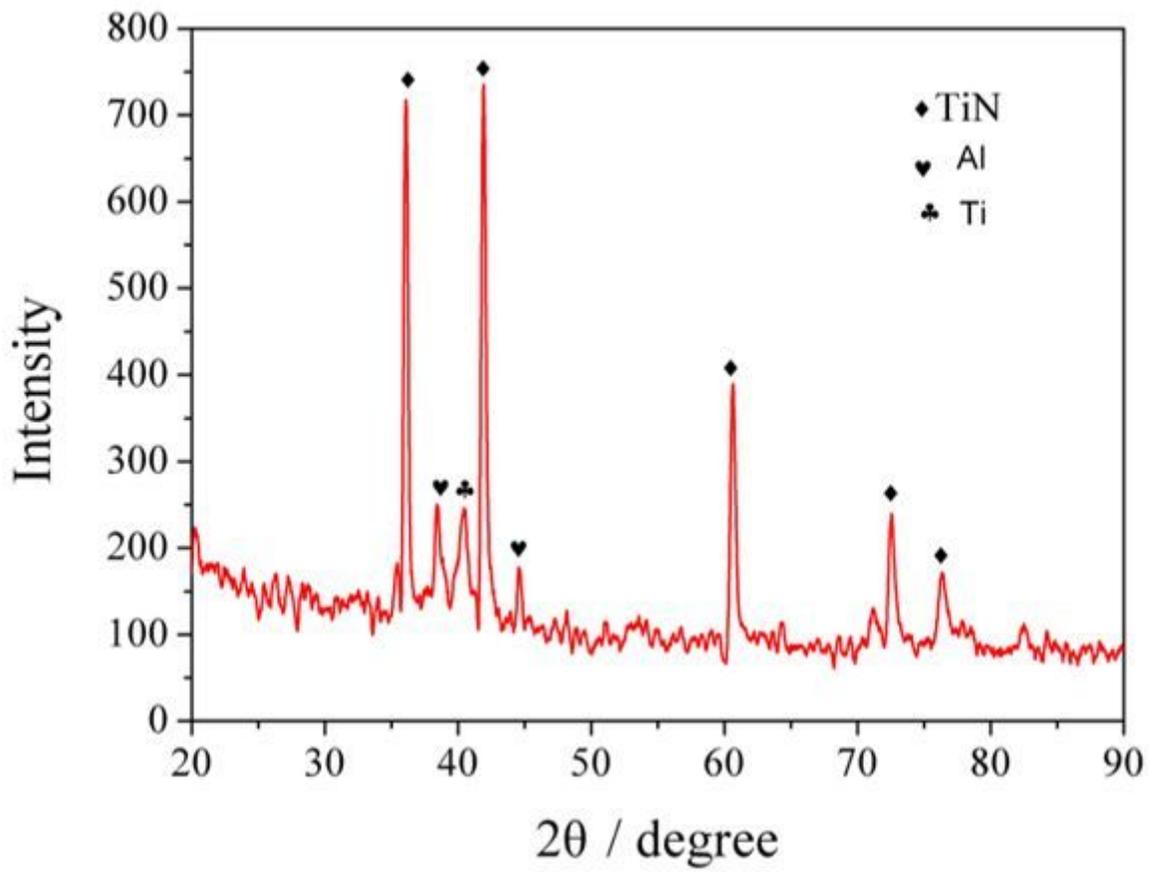


Figure 6

XRD analysis of gas-liquid mixed EDMed surface

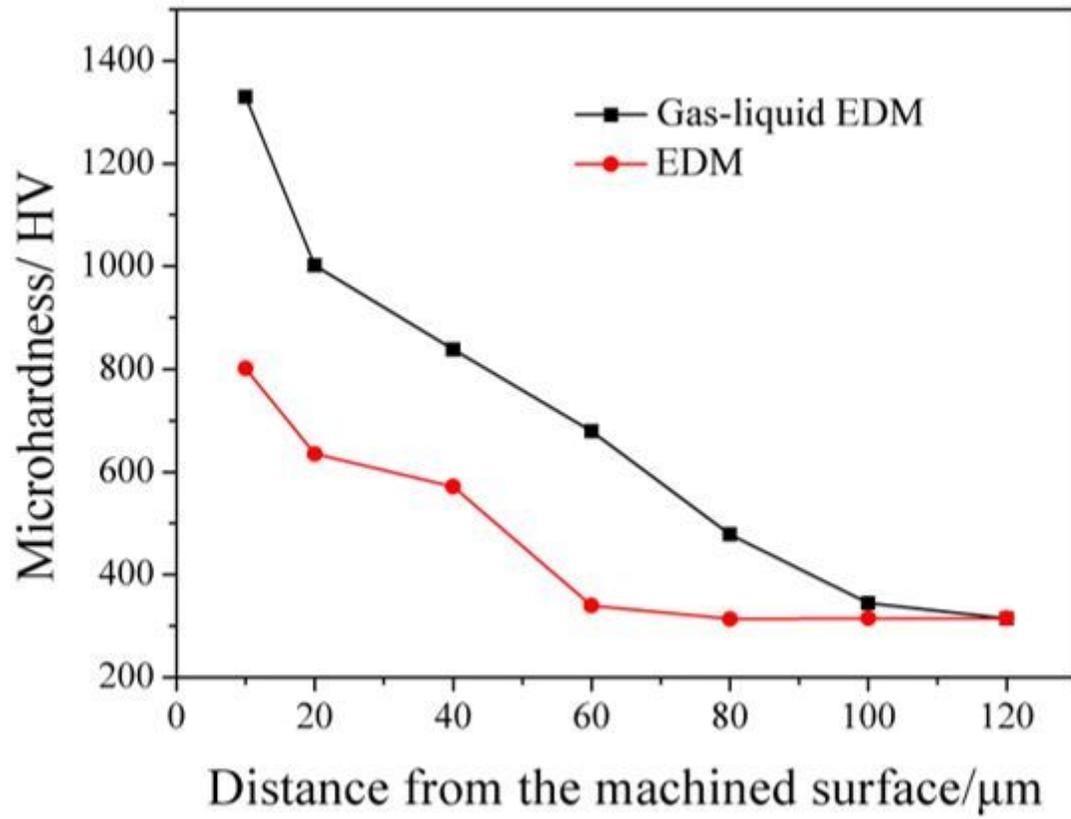


Figure 7

Micro hardness on cross section of machined surface at different dielectric

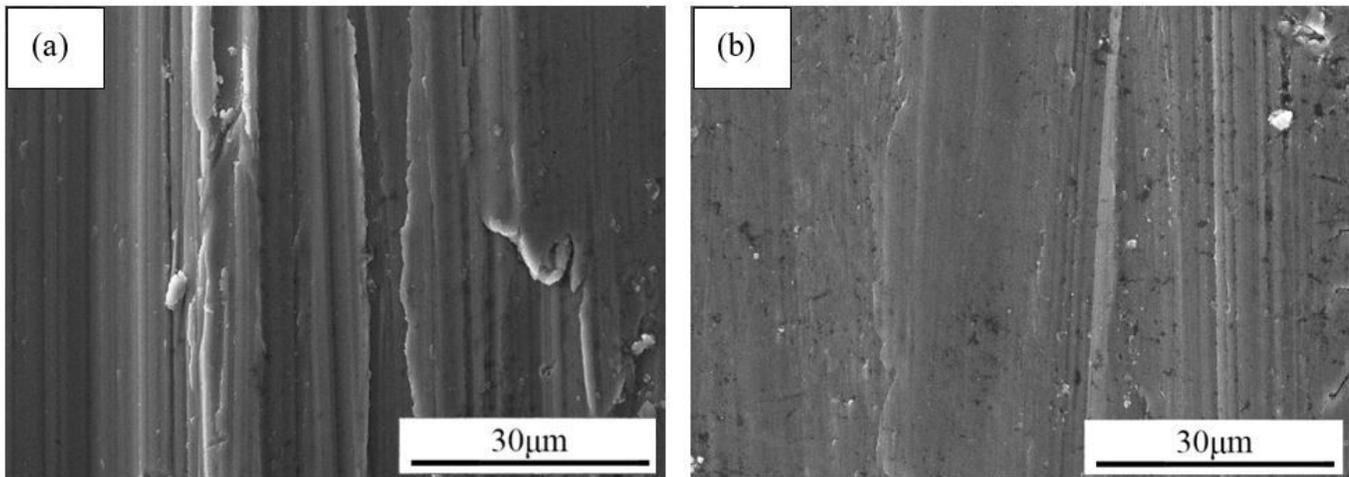


Figure 8

Wear trace surface morphology (a) EDM (b) gas-liquid mixed EDM