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**Influence of Carbon in Iron on Characteristics of  
Surface Modification by EDM in Liquid Nitrogen**

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**ARTICLE INFO**

*Article history:*

Received 23 August 2011  
Received in revised form  
23 September 2011  
Accepted 26 September 2011  
Available online  
26 September 2011

*Keywords:*

EDM,  
Surface modification  
Titanium nitride,  
Liquid nitrogen.

**ABSTRACT**

Many surface modification technologies have been proposed and carried out practically by CVD, PVD et.al. Carbonized layer has been made using EDM method. In this paper, to make the nitride layer by EDM some new trials were carried out using a titanium electrode in liquid nitrogen. Experiments were carried out on carbon steel (S45C), pure iron and cast iron. TiN can be obtained on EDMed surface. Moreover, TiCN can be found on cast iron and steel (S45C) by XRD investigation. To confirm the fabrication mechanisms of modified layer on the steel, the following experimental factors were investigated by EDS.

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## 1. Introduction

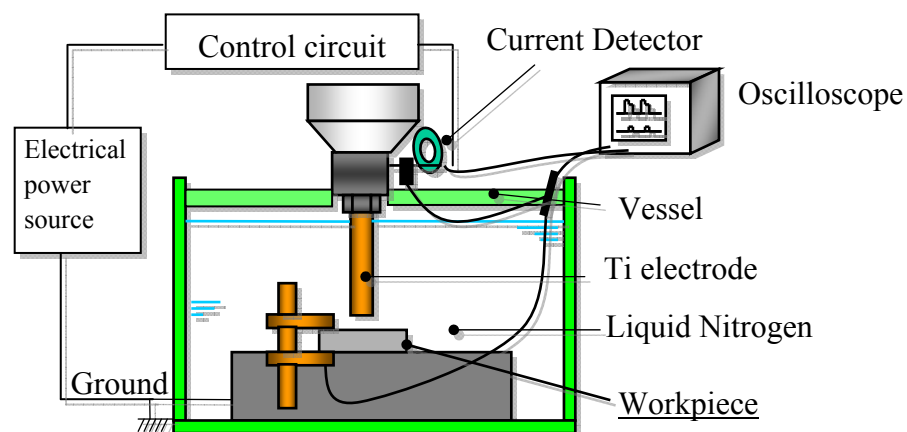
Many surface modification technologies have been proposed and carried out practically by CVD, PVD et.al. Surface modification by EDM have been succeeded to make the modified layer

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i.e. TiC, Si, WC etc. on the work piece by EDM method (N.Saito *et.al.*,1993). In this method, the carbon element that is supplied from the dissolution phenomena of working oil during discharges reacts with the electrode element of Titanium. When the compacted powder body used as an electrode, TiC products piled up easily on the steel surface. On the other hand, the surface modified TiN can be achieved with titanium electrode in liquid nitrogen. (Muttamara *et al.*,2002). Biing Hwa Yan *et al.*, 2005, carried out EDM in urea solution in water with Ti electrode and obtained TiN machined surface. It is interesting that carbon come off by reverse diffusion from the workpiece to the recast layer (Marash et al., 1965). Therefore, the surface modified TiN and TiCN layers have attracted interest for workpiece materials which have high carbon content such as carbon steel and cast iron. Although hardness of TiN layer is lower than TiC layer but friction co-efficiency of TiN layer is quite stable and quite low. In this paper, a new modification method of nitride modified layer on steels by EDM in liquid nitrogen using a titanium electrode is proposed.

## 2. Experimental procedure

Figure 1 shows the illustrated experimental set up. The machining was carried out in liquid nitrogen on carbon-steel (S45C), pure iron and cast iron. Cylindrical Ti solid was applied as an electrode. Table 2 shows chemical composition of S45C. Table 3 shows chemical composition of pure iron and cast iron. The discharge waveforms were observed with a current monitor to analyze the discharge phenomena on this machining.



**Figure 1:** Experimental Set up for EDM in liquid nitrogen.

**Table 1 : Properties - PVD coating Datasheet.**

Coating Material	Colour	Key Characteristics	Hardness (Vickers)	Maximum Working Temperature	Friction Coef (on dry steel)
TiN	Gold	Good general purpose	2300	600C	0.4
TiC	Grey	High hardness	3500+	TBD	>0.1
TiCN	Blue Gray Perple	High hardness, good wear resistance, enhanced toughness	3000	400C	0.4

**Table 2 : Chemical composition of S45C (mass%)**

C	Si	Mn	P	S	Fe
0.45	0.2	0.77	0.17	0.25	Bal.

**Table 3 : Chemical composition of pure iron and cast iron (%)**

Workpiece	C (%)	Si (%)	Fe
Pure iron	<0.005	0	Bal.
Cast Iron	2.11-4.5	3.5	Bal.

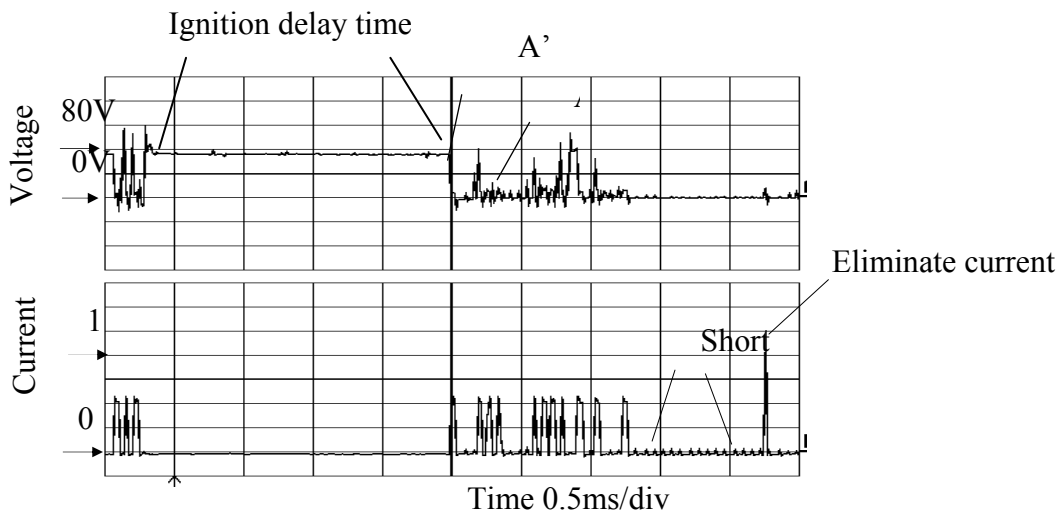
**Table 4 : The experiment conditions**

Parameters	Values
Polarity (Electrode)	-
Current (A)	10, 47
On-time ( $\mu$ s)	32,512
Duty factor (%)	11,50
Open circuit voltage, $u_i$ (V)	220
Water pressure ( $\text{kg}/\text{cm}^2$ )	40
Spindle speed (rpm)	500

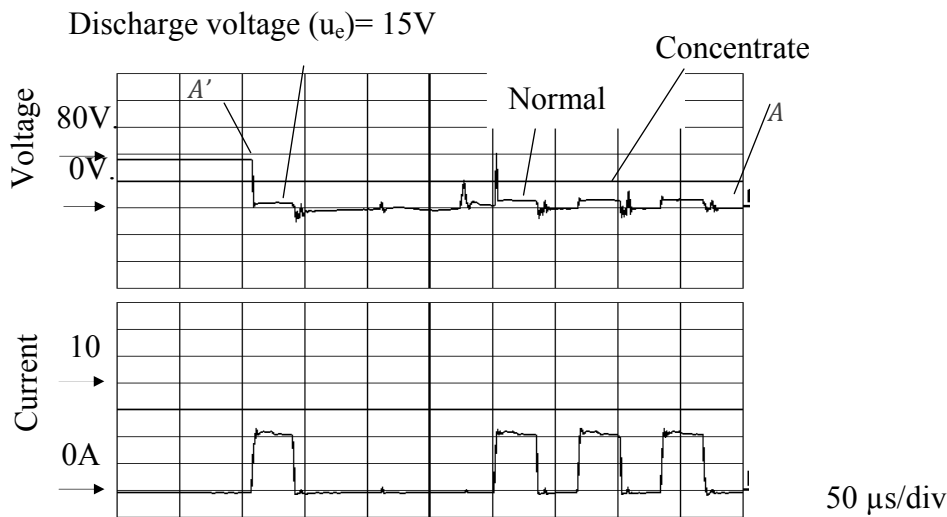
The machining characteristics are estimated in terms of surface roughness, Vicker's hardness, surface layer thickness, X-ray diffraction pattern, EPMA and EDS analysis. The machining conditions are shown in Table 4. The special vessel was designed by polystyrene material for the machining in the liquid nitrogen.

### 3. Results and discussions

At room temperature, liquid nitrogen holds as a boiling state in the vessel. It is known that when the discharge occurs in boiled working medium, the machining phenomena are affected by the bubble generation and the a few discharges contribute to the machining state. Further, exploding the vapor bubble and causing the molten metal to difficult be expelled from the workpiece so that only piling process occurs without machining process. To investigate the pulse discharges in liquid nitrogen, discharge waveforms were observed. Figure 2 shows the discharge waveforms in liquid nitrogen. The detailed waveforms were indicated as A` and A line in Figure 2, are shown in Figure 3. The experiments of EDM were performed on the surface of S45C. Machining conditions were as follows: negative polarity,  $i_e=10A$ ,  $t_e=32\mu s$ , D.F.=50%. There are 4 types waveforms: (a) normal, (b) short, (c) concentrate, (d) short eliminated current. Due to liquid nitrogen holds as a boiling state, therefore EDMed in liquid nitrogen requires a time to break down into ionic (charged) fragments, allowing an electrical current to pass from electrode to workpiece. This region was named as an ignition delay time. Many shorts and concentrate discharges occurred in this process. It can be explained that the sludge was made by the gathering debris phenomena in the gap space during the short circuit and piled on the machined surface during ignition delay time. When the electrode touches the workpiece through the sludge, the concentration of discharge pulse and short circuit occurs. It assumed that the surface modified layer was fabricated by these special discharge phenomena. When short occurs in EDM, it tends to continue long time such as several 100ms from several 10ms. To solve the problem, our EDM system automatically lunches eliminate current to the process (Goto A. et al.,1998). As Figure shows, during off-time it is checked whether gap is short, next pulse is eliminated.



**Figure 2:** Discharge waveforms in liquid nitrogen.



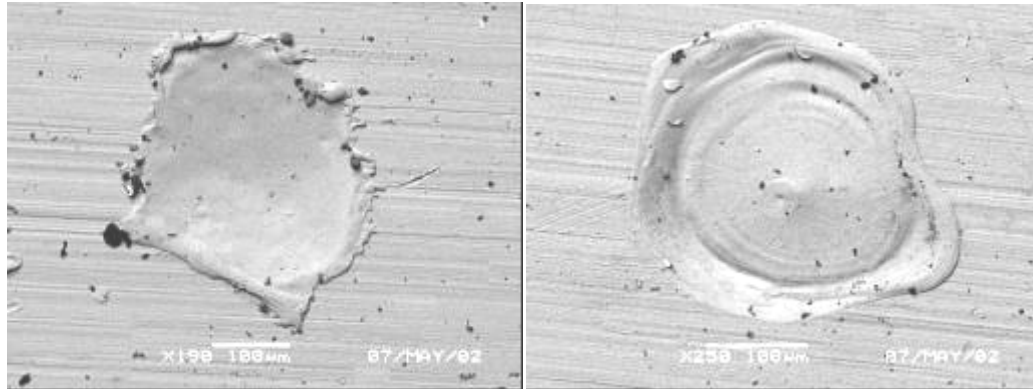
**Figure 3:** Normal discharge and concentrate discharge in period A' – A.

### 3.1 Effect of electrode polarity

On the normal EDM, the positive (+) electrode polarity is chosen for the machining (Janmanee P. and Muttamara A.,2011). On the contrary, the negative polarity (-) often uses for the modified technology (N.Saito *et.al.*,1993), (Muttamara *et al.*,2004), and also machines for insulating ceramic materials (Muttamara *et al.*,2009-2010). These experiments were done under the

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following machining conditions:  $i_e=10A$ ,  $t_e=32\mu s$ , D.F.=11.1% with Ti solid electrode of  $\phi$  5mm. Figure 4 shows the shape of electrical discharge marks from a single pulse electrical discharge experiment in which all other conditions are identical, and only the polarities are changed.



a) Positive

b) Negative

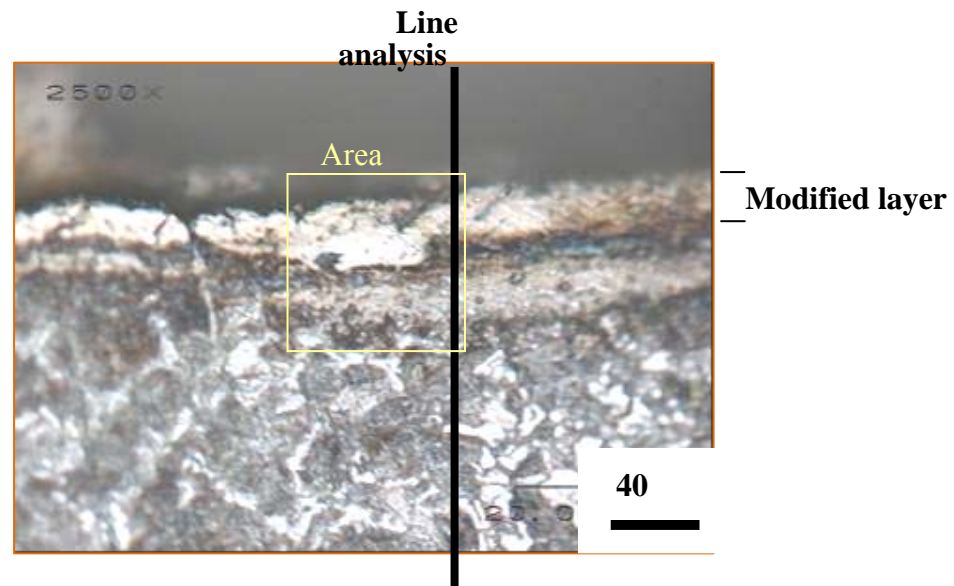
**Figure 4:** Single crater created by a) positive and b) negative polarity.

It can be seen that in the case of negative polarity, large amounts of the melted electrode implant to the workpiece. In comparison, in case of positive, a relatively clean surface crater is formed. Judging from the result, the negative polarity was selected.

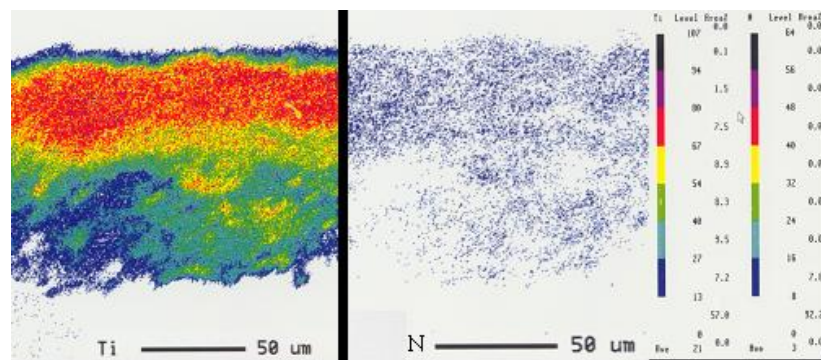
### 3.2 EDM on S45C

To study characteristics of modified layer, the cross sectional of nitride product modified layer on S45C was observed by laser microscope and EPMA analysis. Figure 5 (a),(b) and (c) show the cross sectional EDMed surface by laser microscope, EPMA map analysis and EPMA line analysis of cross sectional EDMed surface, respectively. The golden colored layer could be observed on the machined surface. The characteristics of the modified layer were investigated by the micro-hardness Vickers using a load of 10gf and the EPMA analysis. Figure 6 shows micro-hardness distribution on the cross section of modified layers with solid and semi-sintered. ( $i_e=10A$ ,  $t_e=32\mu s$ , D.F.=11.11%). On the machining of Ti solid electrode, there were three areas: (1) nearest surface region, 0-50  $\mu m$ , the hardness reached to 1300HV that corresponded almost to the same value of other report (table 3.1), (2) thermal affected region, 50-100  $\mu m$ : similar hardness of martensite structure of 800HV, (3) original substrate region: over 100  $\mu m$ . On the contrary, the hardness of region (1) became the same value, 800HV at region (2) on the machining of semi-sintered electrode.

EPMA analysis of Ti, N and C, was carried out on the cross sectional modified surface. The distribution of Ti and N element was divided to three regions same as Figure 5. The distribution of Ti and N element was detected from region (1) to (2). It indicated that the region composed with the thermal affected structure of substrate and the diffused TiN products. In the (1) and (2) region, the higher carbon element was observed than matrix regardless no supplying source around discharge circumstances. Because carbon was observed on the modified layer on S45C. It was thought that carbon come off by reverse diffusion (Barash *et al.*, 1965).

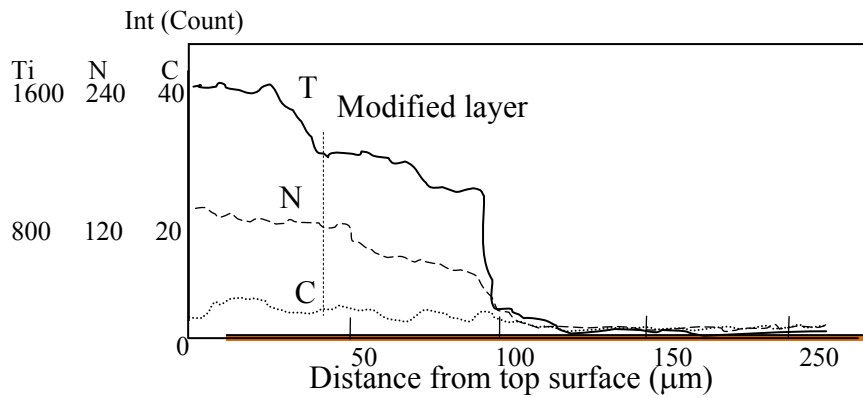


a) Modified layer on S45C in liquid nitrogen



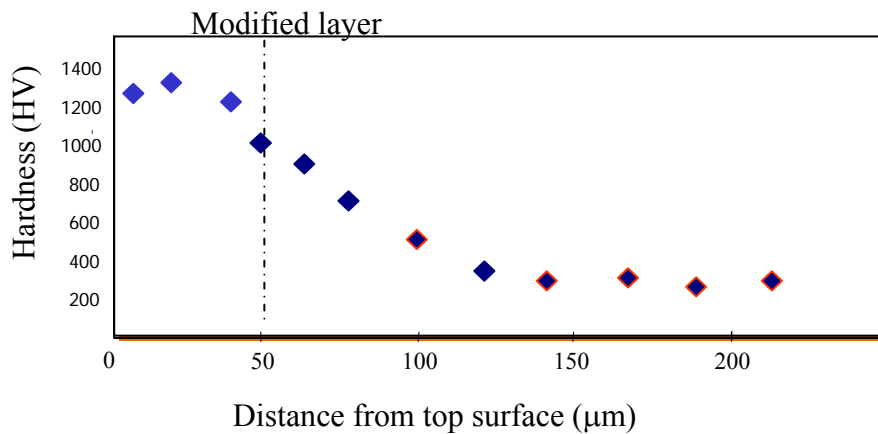
b) EPMA Map analysis Modified layer on S45C in liquid nitrogen

**continue Figure 5 on next page**



c) EPMA Line analysis

**Figure 5:** Cross sectional image of TiN layer on S45C by a) Laser Microscope  
b) EPMA Map analysis c) EPMA Line analysis.



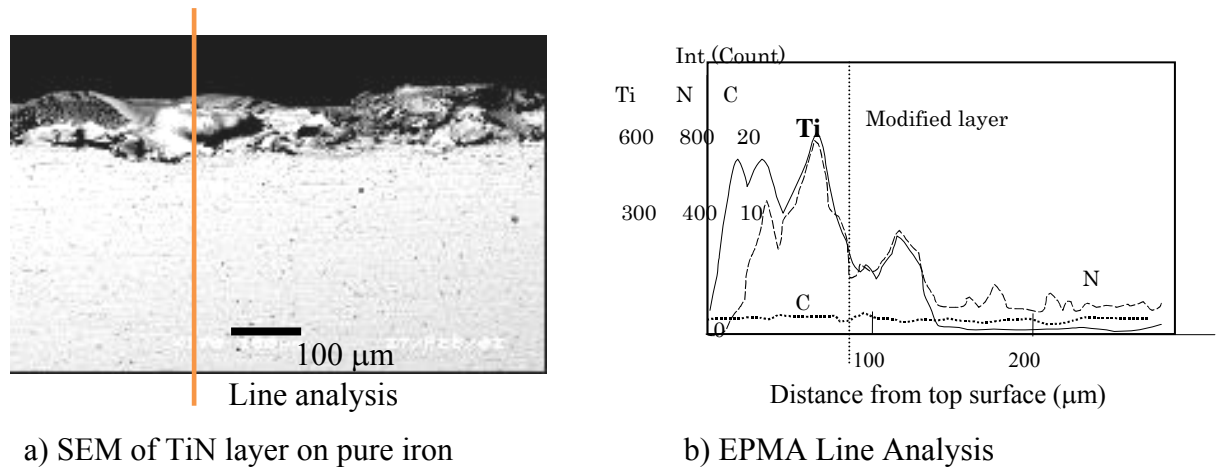
**Figure 6 :** Relationship between micro-hardness Vickers against the cross section of modified layer on S45C.

### 3.3 EDM on Pure iron

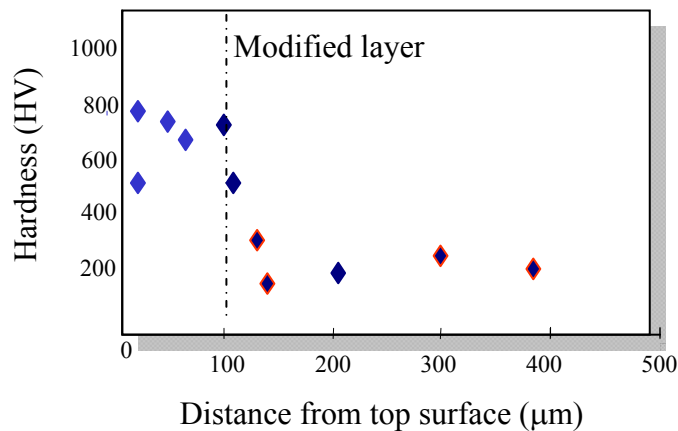
Pure Iron does not contain carbon (less than 0.005%). The concentration of substances on the cross section of modified surface on pure iron were carried out with Ti solid. Figure 7 shows cross sectional SEM of EDMed surface on pure iron compared with EPMA results. Figure 8 shows the sectional micro-hardness measurements of modified surface. The thickness of modified layer is 100 µm as same as the modified layer on S45C. From the sectional micro-hardness result,



hardness of modified surface is 600-800 HV. The hardness of modified layer on Fe is lower than that on S45C. This is considered that carbon in the material of S45C affect to the compound of modified layer.



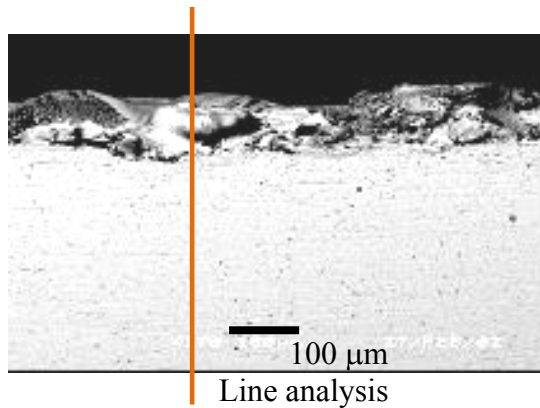
**Figure 7:** Cross sectional TiN layer on Fe a) SEM and b) EPMA Line Analysis



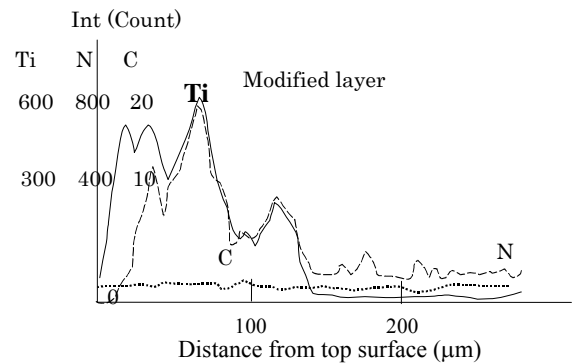
**Figure 8:** Micro-hardness distribution (EDM Conditions;  $i_e=47A$ ,  $t_e=256\mu s$ , D.F.= 11.1%).

### 3.4 EDM on cast iron

Cast iron was used to confirm (reverse) diffusion of carbon. In this experiment, discharge current ( $i_e$ )=47A, discharge duration ( $t_e$ )=256 $\mu s$ , (D.F.)=11.11%, were selected for EDM condition. Figure 9 shows cross sectional SEM of EDMed surface on cast iron compared with EPMA results. Figure 10 shows the sectional micro-hardness measurements of modified surface.

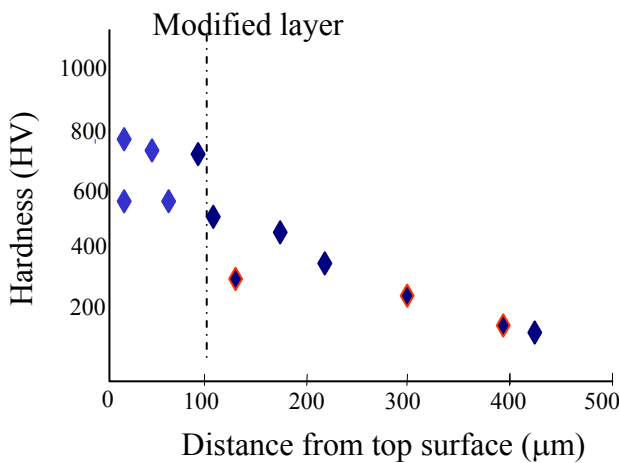


a) SEM of TiN layer on cast iron



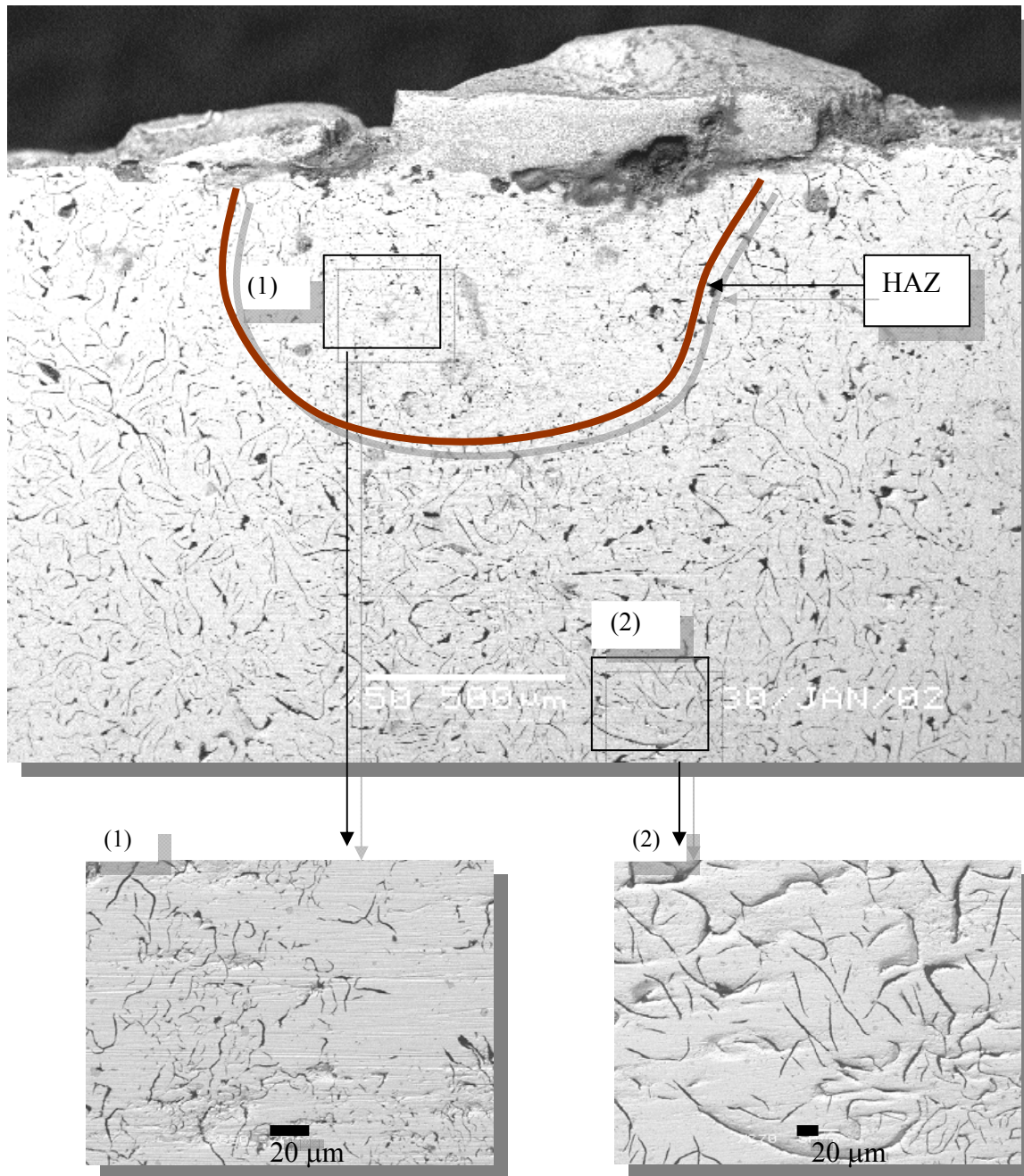
b) EPMA Line Analysis

**Figure 9:** Cross sectional TiN layer on cast iron a) SEM and b) EPMA Line Analysis



**Figure 10:** Micro-hardness distribution (EDM Conditions;  $i_e=47A$ ,  $t_e=256\mu s$ , D.F.= 11.1%)

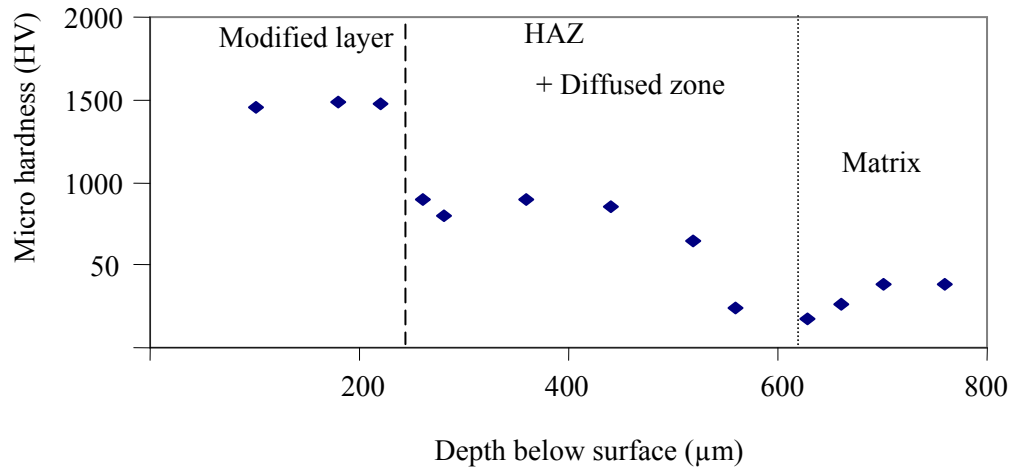
The C and N elements concentrations are measured on the modified layer, distance of the generation of C and N elements are 250  $\mu m$  of modified layer as can be seen from the Figure 9. First, it should be noticed that system experiment was decarburizing. So carbon on modified layer should come from the precipitated graphite in the cast iron. However, we cannot see clearly on EDS result of carbon. Etching was done on cross section surface of cast iron as shown in Figure 11.



**Figure 11:** SEM micrographs of etched cross section surface of cast iron

The low part represents the base material, the central part in the curve mark represents the base material that effect from heat affected zone (HAZ), and carbon diffused zone. The modified layer was generated irregularly. The dendritic parts in substrate are graphite exist in the form of flakes. It is pointed out that some areas inside close line carbon are depressed. The large scale of structure

under modified surface is shown in Figure 11 (a) Also the structure of normal graphite in cast iron is shown in Figure 11 (b). The presences of graphite in HAZ (a) are different from normal content (b). Therefore, it is considered that precipitated carbon diffuses by discharges or the changing of structure of case iron.

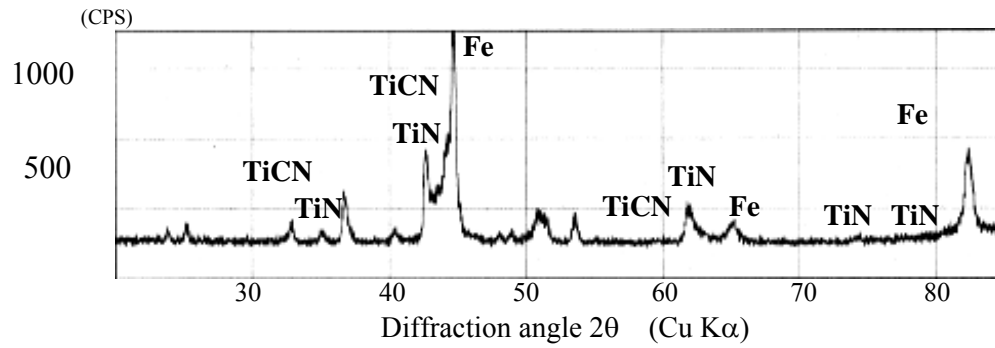


**Figure 12:** Section hardness of machined surface before and after annealing

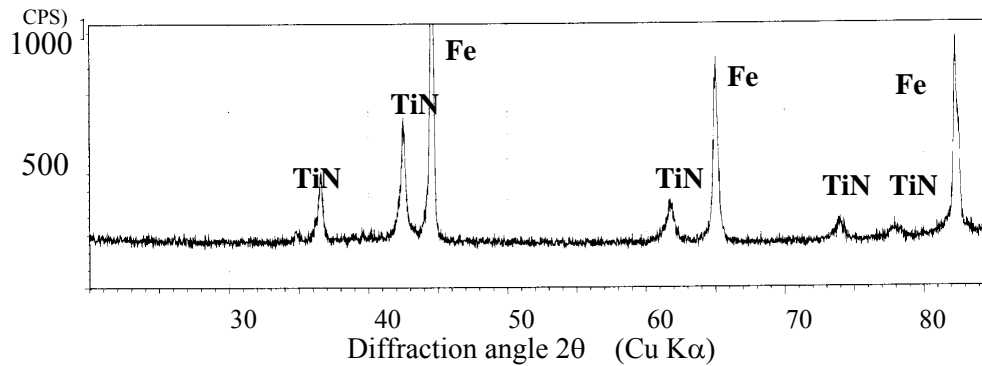
To investigate effect of carbon and HAZ on the hardness, the hardness was evaluated on cross sectional of cast iron. Figure 12 shows the sectional hardness measurement of modified surface on cast iron. It can be considered that the machined surface is covered with TiN and TiCN layer. The hardness of modified layer is about 1450 Hv. On HAZ region, the hardness decreases gradually according to the distance from the surface. It reaches to the hardness of matrix cast iron through that of re quenched region. Some hardness regions on HAZ are below the hardness of matrix region, it is considered that the coming off of carbon effects to the hardness of that region. HAZ.

### 3.5 X ray-diffraction (XRD) analysis

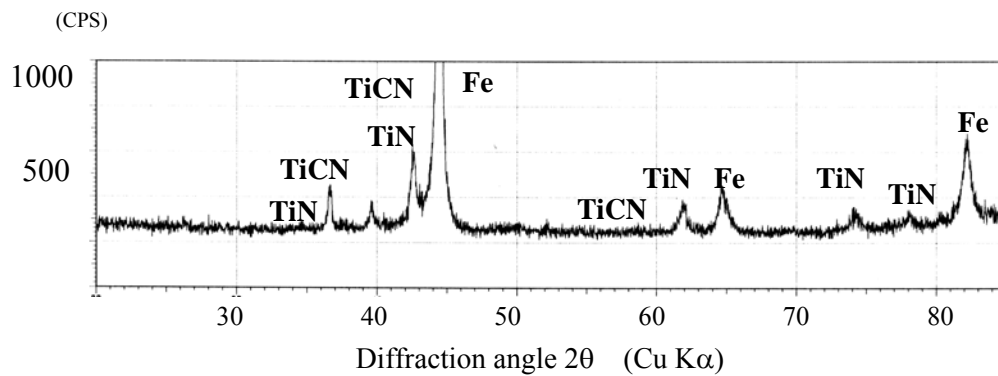
As mentioned above, the some modified layer could be adhered on the work piece by EDM in liquid nitrogen. To confirm the layer composition X ray-diffraction (XRD) pattern was investigated for the EDMed surface with Ti solid electrode. Figure 13 shows the result of XRD on EDMed surface on S45C compared with EDMed surface on pure iron and cast iron. The peak of TiN and TiCN are very near. From the EPMA results and the hardness results, it indicates that the EDMed surface on S45C and cast iron are composed of TiN and TiCN. On the other hand, only TiN layer was observed on the EDMed surface of pure iron.



a) EDMed surface on S45C



b) EDMed surface on pure iron



c) EDMed surface on cast iron

**Figure 13:** X-ray diffraction patterns obtained from the EDMed layer in liquid nitrogen by solid Ti on a) S45C , b) pure iron and c) cast iron.

## 4. Conclusion

A new EDM surface modification method was tried in liquid nitrogen on S45C steel in various conditions. The results were summarized as follows:

- (1) In liquid nitrogen, machining process is not obtained, but the TiN products adhere on the work piece.
- (2) Ti and N element diffused from nearest surface to the thermal affected zone.
- (3) Discharge causes carbon migration from deeper layers of the substrate.
- (4) TiCN modified layer could be generated on carbon steel and cast iron because carbon from substrate diffused to modified layer and reacted with nitride product of modified layer.

## 5. Acknowledgement

The authors are grateful to Faculty of Engineering, Thammasat University, the National Research Council of Thailand (NRCT), the Thailand Research Fund (TRF) and the National Research University Project of Thailand Office of Higher Education Commission for the research funds and T. Klaykaow for carrying out this work.

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