

Enhancement of Case Hardness in AISI1025 Steel at Low Voltage Micro Arcing in a Liquid Solution

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Abstract

Micro arcing developed at an electrode having smaller surface area as compared with that of the other electrode of an electrolytic cell when the cell was supplied DC voltage of around 61volts. AISI1025 steel specimens of 20mm diameter and 50 mm length were treated in an electrolytic cell with an aqueous solution of 100ml glycerin and 100gmNH₄Cl. Voltage applied to the cell was gradually raised from 0 to 110 Volts. Current-voltage data was acquired using data acquisition card. Temperature measurement was done using chromel_alumel thermocouple. Hardness measurements were done using Vickers hardness tester. Microstructural characterization and elemental analysis was done by optical and SEM techniques

Keywords: micro arcing, EPP, elemental diffusion, SEM

1 Introduction:

Heat treatment has been undergoing rapid changes in recent years. Many, if not all, of these changes are brought about due to the stringent restrictions being placed by environmental considerations and increasing cost of power. Surface engineering on the other hand has been assuming steadily increasing importance by virtue of developments in other technologies such as generation of intense laser beams, production and control of widely different ion beams, greater resolutions in scanning probe microscopes etc.

The plasma was formed on the surface of an electrode in a liquid solution when a high voltage applied to the electrodes and high voltage electrolysis was carried out. A real time heat calibration system was designed and used to measure the temperature developed during such high-voltage electrolysis. It was seen that the temperature developed was in excess of the power input. Heat generation process depended on the conditions for the electrolysis. There was no excess heat generation in the beginning of the high-voltage plasma electrolysis but the heat generation exceeded when the plasma electrolysis was performed for a longer time. 100% reproducibility would be achieved if all factors such as temperature, voltage and duration are optimized [1]. Tadahiko Mizuno observed some unique reactions occur at the electrode surface. Such reactions may not occur at energy levels available during electrochemical analysis. The heat generation can be achieved by the plasma electrolysis and the plasma phenomenon has been reported for a long time in the literature [2].

Discharge phenomenon associated with the plasma electrolysis has been discovered centuries ago by Sluginov. These discharge phenomena were studied in detail in 1930 by Gu'nterschultze and Betz [3]. Though the discharge phenomena were discovered a lot earlier, their practical uses were not exploited till 1960. This time around McNeill and Gruss achieved deposition of Cd-Neobdate onto the cadmium anode by using discharge phenomenon in a Niobium containing solution [4]. And this was the beginning of exploring the practical applications of plasma discharge. Now the researchers round the world began exploring the practical benefits of plasma discharge. Markov and coworkers achieved deposition of oxide on the aluminum anode by

using the same discharge phenomenon [5]. During 1980s, a number of researchers from Russia, Germany, US and China began studying in more detail the possibility of using the electrolytic discharge for the deposition of oxide layers on the anode surface. The researchers who studied these diffusion techniques in more detail are the Markove and coworkers [6], Kurze and coworkers [7-9], Fyodorov et al [10], Gordienko and coworkers [11], Kurze and coworkers [12]. Early industrial applications were introduced by Kruz and coworkers in Germany.

R.J. Gradkovsky, S.N. Bayles, researchers from USA and the W. Xue, Z. Deng, Y. Lai, R. Chen, J. of China worked in the same area [12, 13]. Initially the information available on the process phenomenology was sparse and also there was a lack of understanding regarding the process. So the same technique was called by different names like anode spark electrolysis, micro plasma oxidation, plasma electrolytic anode treatment etc. In fact all these techniques were nothing but the same i.e., plasma electrolytic oxidation (PEO).

2 Experimental Setup

A plastic beaker (5-liter) to contain the liquid electrolyte solution was used. Two rectangular Perspex bars were vertically placed at the bottom of this beaker to support the anode ring. A copper clip was used as a specimen holder. The specimen holder was firmly held by a rectangular Perspex bar, which could be centrally lowered into the beaker. Figure 1 shows the schematic of the set up and figure

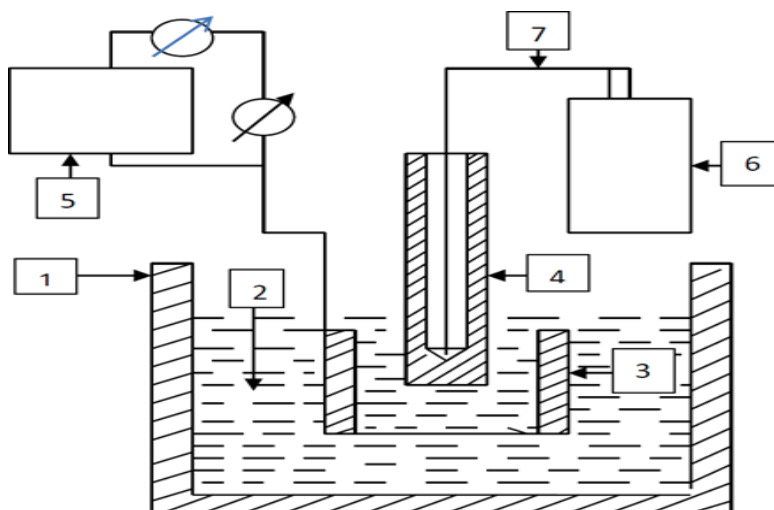


Figure 1: The experimental set up used to heat the steel specimens by micro arcing in aqueous solution. 1. Plastic pot, 2. Electrolyte bath, 3. Passive electrode, 4. Active electrode, 5. Power supply, 6. Data logger, 7. Thermocouple

Finely ground AISI-1025 cylindrical steel specimen was made cathode of the cell. A stainless steel ring of 100 mm diameter and 20 mm height was used as an anode of the cell. Electrolyte bath used was consisted of the 3 liter of distilled water in which 100 ml glycerin and 100 gm NH_4Cl was added. DC power supply (0-250V and 20A) was used and the treatment was done for 10 sec, 30 sec, 1 min & 5 min. Chromel-alumel thermocouple was used for temperature measurements.

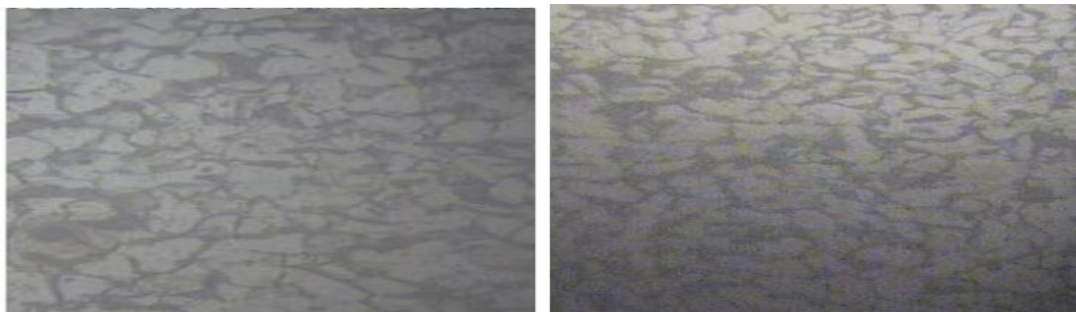
Microstructural characterization was done using optical and scanning electron microscopy and the hardness measurements were done using Vickers hardness test machine.

3 Experimental Method

Cylindrical steel specimens of AISI1025 of 20mm diameter, 50mm length were finely ground to a smooth surface finish and then heated to a temperature of 850 0C in an electrolyte bath of the 3 liter distilled water in which 100ml glycerin and 100 gm NH4Cl was added. DC voltage applied to the cell was gradually increased from zero to 61 Volts. Heating of the specimen was performed for 10 sec, 30sec, 1 min & 5 min. After this heating time the power supply to the cell was cut-off. The specimen was taken out and its microstructural characterization and hardness measurement was done.

4 Results And Discussion

As the treated specimens were not quenched, it showed no change in the microstructure but exhibited rise in hardness. Hardness of the steel specimens increased from 214 VPN to 228VPN when the specimens were treated for duration of 1 min. Hardness values were found to increase from 214 to 235 VPN when the specimens were treated for longer duration of 5 min. Figure 2 and 3 shows the microstructure of the sample before and after the treatment.



500X 500X

Fig.2 Microstructure before treatment **Fig.3** Microstructure after treatment

Hardness measurements were performed using Vicker's hardness tester. Applied load was 60gm and the indenter used was the diamond pyramid. Table 1 shows the hardness variation before and after treating the sample

Table 1: Hardness variation on diffusion of carbon atoms into steel sample.

Sample	Time of treatment	Hardness	Hardness
		before treatment	after treatment
AISI-1025	05 min	214 VPN	235VPN
AISI-1025	01 min	214VPN	228 VPN

Microstructure has not undergone any significant change. It means that sample was not cooled properly and the sufficient cooling rates required for the martensite transformation has not reached. However, increase in hardness from 214VPN to 235VPN indicated that carbon from the electrolyte bath has diffused into the steel surface. Hardness of steel increases with increase in carbon content. SEM and EDAX study of treated specimens Carburized steel specimens were dry polished on SiC coated emery papers of grades 1/0, 2/0, 3/0, and 4/0. The rough polished specimens were then fine polished on a single disc fine polishing

machine. Al₂O₃ powder particles suspended in tap water was applied on the rotating velvet cloth covered disc of the fine polishing machine and then specimens were fine polished under moderate pressure. Topographic images of the prepared specimens were obtained by using SEM. Topographic images has revealed the carburized layer which appears differently than the inner core of the specimen as shown in the figure 4.

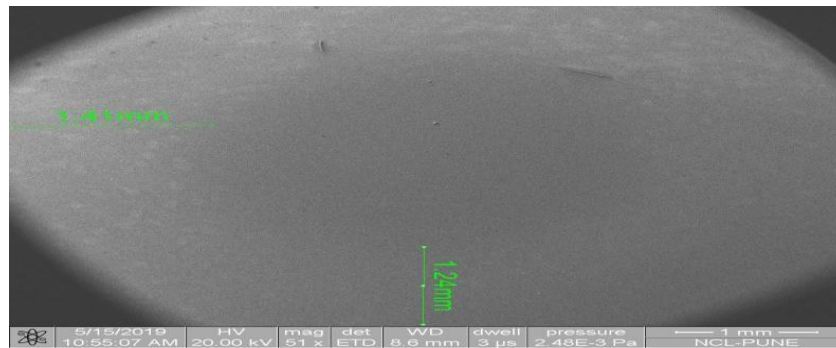
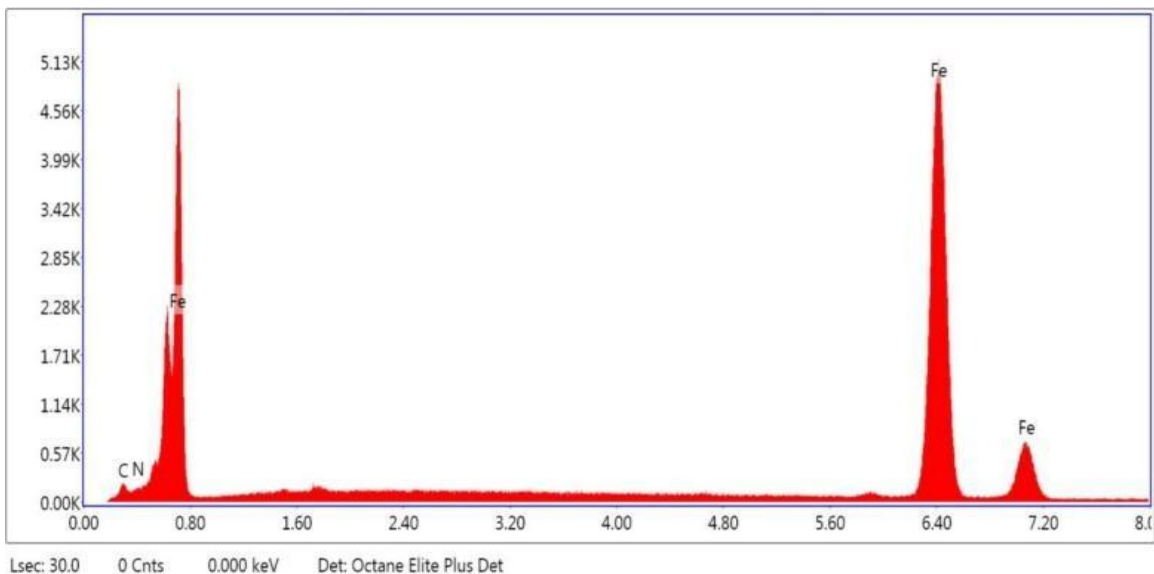


Fig.4 -SEM topographic image of carburized specimen obtained at 20.00kV, treated for 5 mins revealing carburized zone(1.24 to 1.41mm) and central noncarburized region.

Specimens treated for the duration of 5 mins showed that elemental diffusion of carbon has occurred when the steel was subjected to electrolyte plasma processing by cathodic plasma regime. The carbon enriched region formed is clearly revealed in the SEM micrograph obtained at 20 kV. Carburized depth reached within 5 mins was found to be 1.41mm and 1.24mm at some other places. Uniform case depth has not been observed.

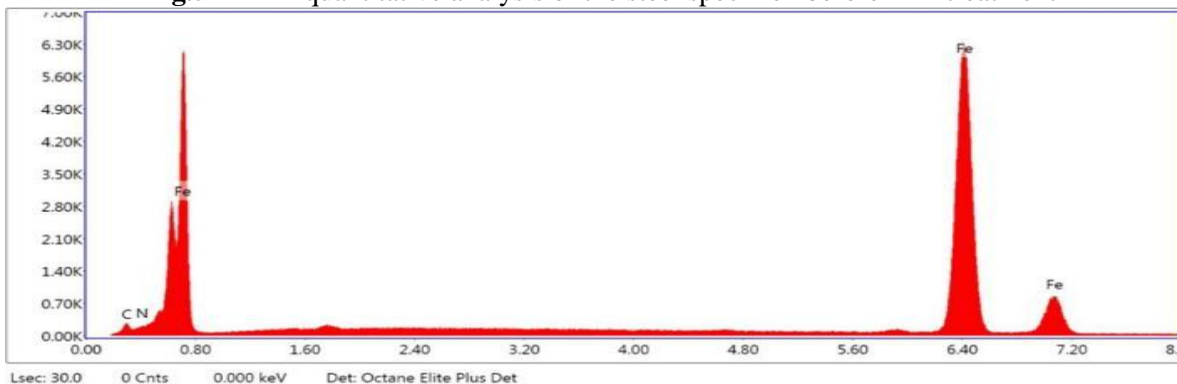
Elemental diffusion into steel surface has been investigated by doing EDAX quantitative analysis of the treated samples. EDAX quantitative results are shown in the figure 5 & 6. It has shown that carbon



enrichment of the steel surface is achieved.

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
C K	0.27	1.25	3.12	78.84	0.0007	1.3196	0.8355	0.2002	1.0000
N K	0.00	0.01	0.03	99.99	0.0000	1.2931	0.8485	0.2653	1.0000
FeK	99.73	98.74	4629.75	1.80	1.0205	0.9990	1.0004	1.0001	1.0242

Fig.5 EDAX quantitative analysis of the steel specimen before EPP treatment



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
C K	0.38	1.75	5.43	72.93	0.0010	1.3191	0.8357	0.2004	1.0000
N K	0.00	0.01	0.03	99.99	0.0000	1.2926	0.8487	0.2643	1.0000
FeK	99.62	98.25	5742.10	1.76	1.0190	0.9986	1.0005	1.0002	1.0242

Fig.6 EDAX quantitative analysis of the steel specimen after EPP treatment, indicating rise in carbon to 0.38Wt%.

Electrolyte plasma processing is so fast and efficient that carbon diffusion into steel surface becomes possible in short period of time. In just 5min, steel surface enriched with carbon and concentration increased to 0.38Wt%. Diffused carbon concentration increased with increase in time and voltage applied to the cell.

5 Conclusion

1. It was feasible to achieve carbon enrichment of low carbon steel surface by EPP treatment at the applied voltage of 61V and above when processing was done in an electrolyte bath of glycerin and ammonium chloride. Carbon enrichment of steel surface was achieved within 5mins.
2. SEM micrographs and EDAX analysis performed has presented clear evidence of carbon enrichment of steel surface via elemental diffusion of carbon. EPP system can be automated for more effective and varied applications

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