



## QUENCHING AND PARTITIONING OF AISI 4340 STEEL

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### Abstract

Quenching and Partitioning (Q & P), an innovative heat treatment process was developed by J. G. Speer in 2003 to produce third generation high strength steels with improved toughness for making chassis of high speed cars. The Q & P process was applied to the low alloy AISI-4340 steel for different partitioning time periods. The characterization process encompassed microscopic study through optical microscope and hardness testing by micro Vickers hardness tester. Also, corrosion behaviour of different samples, partitioned at various time periods was studied by Gamry Potentiostat. Light optical microscopy revealed that microstructure consists of lath martensite and inter lath blocky retained austenite. The volume fraction of the retained austenite showed an initial progressive increase in proportion with the partitioning time but later pursued a decrease at partitioning time of 60 and 120 sec steadily. The hardness values were increased from 236 HV for a non-heat treated sample to 477 HV for 15 sec partitioned sample thereafter decreased with increase in partitioning time. The electrochemical test results showed a continued mitigation in the corrosion rate from 3.581 mpy for non-heat treated sample to 0.08 mpy for the 30 sec partitioned steel sample.

**Keywords:** Quenching, Partitioning, Corrosion, Vickers, Microstructure, Martensite

### 1. Introduction

The approaching challenges to develop high strength chassis design of automobiles with weight reduction for an optimized mechanical performance are gradually increasing in this era of technological innovation. The advanced high strength steel (AHSS) came into being to offer having tremendous combination of strength, toughness, formability and fatigue resistance in a continual progressive mode [1]. The first generation of AHSS Steels such as dual-phase (DP) [2] steels and transformation-induced plasticity (TRIP) steels [3,4], complex phase (CP) and martensitic steels (MART) were developed. After that twinning induced plasticity (TWIP), aluminium added light weight steels having induced plasticity and shear band strengthened steel (SIP) are known as second generation AHSS were developed. However in order to achieve profound improvement in mechanical strength and toughness, over the past century, scientists have been able to design third generation of AHSS with unprecedented strength and toughness better than previous generations and at significantly lower cost [1,5]. With a supporting edge, a new heat treatment process known as quenching and partitioning was proposed by J. G. Speer [6] in order to achieve greater strength and toughness than the first and second generation at lower cost [5] Eventually, a new heat treatment process proposed by J. G. Speer introduced as quenching and partitioning of micro-phases of steel was adopted in practice [7].

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After the success of this process many researchers applied this technique within their research works. In 2003, J. G. Speer et al. studied carbon partitioning into austenite after martensite transformation [8]. In 2007, A.J. Clarke et al. worked on carbon partitioning to austenite from martensite or bainite in the Q & P process [9]. In 2008, M.J. Santofimia et al. used Q & P process in characterization of microstructure in low carbon steel [10]. In 2009, A.J. Clarke et al. studied the influence of kinetics of carbon partitioning to final austenite fraction during Q & P process [11]. In 2010, L. Kucerova et al. studied 42SiCr steel after doing Q and P by laser scanning confocal microscopy [12]. In 2012 E. P. Bagliani et al. performed quenching and partitioning process on medium carbon steel to study its microstructure, toughness and tensile properties [13]. In 2013, X. Tan et al. studied microstructure-properties relationship in a one-step quenched and partitioned steel [14]. In 2014, X. Han et al. applied hot stamping process by integrating Q & P process to improve mechanical properties [15]. Structure obtained by Q & P process consisted of martensite with retained austenite, called duplex microstructure [16]. Nickel and Manganese are austenite forming elements and they are added to steels to get retained austenite [17]. The steel selected for Q & P process must contain either high Nickel contents or high Manganese contents. AISI-4340 containing high Nickel contents and hence, was selected for this research work and Q & P process was applied to it. Microstructures were characterized by Light Optical Microscope. Vickers hardness and corrosion behaviour became an integral part of this research work.

**2. Materials and Methods**

*2.1. Materials*

AISI-4340 steel grade was selected due to high Nickel content required for Q & P process. Its chemical composition is given in Table 1. This steel was obtained from the Peoples Steel Mill Ltd. Karachi in rod shape.

**Table 1: Chemical Composition (wt. %) of AISI-4340 steel studied.**

Steel	C	Si	Mn	Cr	Mo	Ni	Cu	Fe
AISI 4340	0.422	0.283	0.719	0.785	0.216	1.736	0.257	Balance

The steel rods having diameter of 32 mm were cut into lengths of 6 inches on a power Hacksaw. Then, these small rods were further cut down into discs of 1cm thickness by using a precision disc cutter. Finally, disc shape samples were cut into four pie shape sections. The samples were cleaned in aqueous solution of 5% Na<sub>2</sub>CO<sub>3</sub> at 60 °C for 10 minutes to remove grease, oil, dust particles and other contaminations.

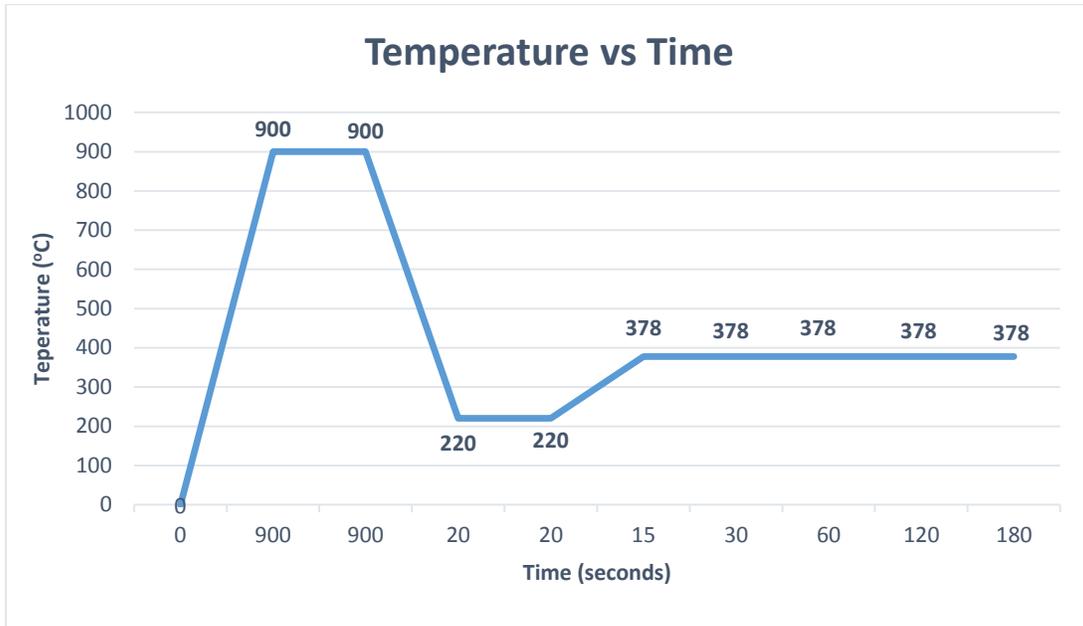
*2.2. Heat treatment*

Austenitising temperature was selected to be 900 °C. For quenching, martensite-start temperature (Ms) was calculated 296 °C using Nehrenberg’s equation [18].

$$Ms = 498.9 - 300C - 33.3Mn - 22.2Cr - 16.7Ni - 11.1(Si+Mo) \text{ ----- (1)}$$

Quenching and Partitioning temperatures were 220 °C and 400 °C, respectively. The samples were austenitised at 900 °C for 900 sec and quenched in salt-bath (50%KNO<sub>3</sub>+50%NaNO<sub>3</sub>) in a crucible furnace maintained at 220 °C for 20 sec. After 20 sec, the samples were partitioned in another salt-bath (50%KNO<sub>3</sub>+50%NaNO<sub>3</sub>) in

crucible furnace maintained at ~400 °C for partitioning times of 15, 30, 60, 120 and 180 sec. After partitioning the samples were air cooled and rinsed with water to remove any attached salt. The applied Q & P heat treatment cycle is shown in Figure 1.



**Figure1:** Graph showing heat treatment cycle of Q&P process applied to AISI-4340 low alloy steel.

### 2.3. Metallography

The heat-treated samples were mounted in Bakelite powder using Buehler brand model Simplimet 1000 mounting press for metallography. Grinding of mounted specimens was carried out by following standard procedure and practice using FEPA grade grinding papers.

Rough polishing was performed by using 3 and 6 micron diamond pastes on Nylon cloth, while fine polishing was carried out using 1 and 0.25 micron diamond pastes on velvet cloth. The polished samples were etched in 2% Nital for 10 sec. Light optical digital images of the etched samples were taken at a magnification of 500X using Leica brand metallurgical microscope.

### 2.4. Hardness Testing

Vickers hardness testing of the steel samples was performed on Micro Hardness Tester by applying a load of 500 gm (4.903N) for 10 sec.

### 2.5. Corrosion Testing

Electrochemical behaviour of quenched and partitioned AISI-4340 steel was studied by using Gamry Potentiostat in 3.5% NaCl solution. Saturated Calomel Electrode (SCE) was set as reference electrode and the auxiliary electrode was made up of graphite. AISI 4340 steel samples were soldered with copper wires and mounted in epoxy resin to form working electrodes.

The samples were pie shaped  $\frac{1}{4}^{\text{th}}$  of a circle. Hence, exposed surface area was calculated by using the equation 2;

$$A = \frac{1}{4} \pi r^2 \text{ ----- (2)}$$

Calculated exposed surface area of the samples and potentiodynamic scan parameters are given in the Table 2 and 3, respectively.

**Table 2: Calculated exposed surface areas of the samples.**

Samples ID	Radius (cm)	$A = \frac{1}{4} \pi r^2$ (cm <sup>2</sup> )
WHT (without heat treatment)	1.60	2.01
15	1.45	1.65
30	1.50	1.766
60	1.50	1.766
120	1.55	1.88
180	1.45	1.65

**Table 3: The parameters of potentiodynamic scan on Gamry Potentiostat.**

Parameters	Values
Initial potential	-0.5V vs E <sub>oc</sub>
Final Potential	+1.5V vs E <sub>oc</sub>
Time	1s
Scan rate	5 mV/s
Density	7.83377 g/cm <sup>3</sup>
Eq. weight	27.802 g

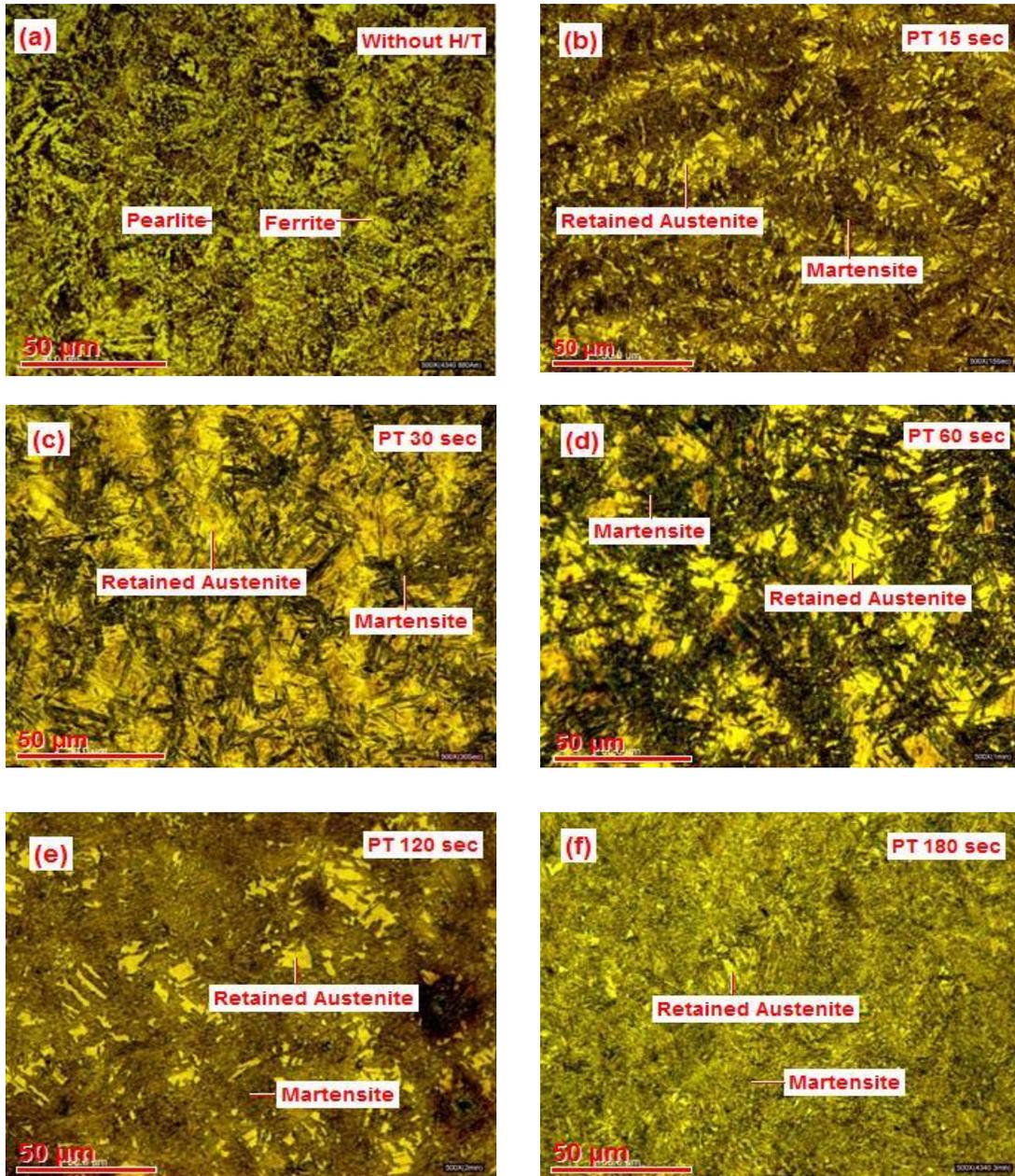
### 3-Results & Discussion

#### 3.1 Microscopic Examination

As cast AISI-4340 steel consisted of lamellar pearlite uniformly distributed in the matrix of bcc ferrite, as shown in Figure 2 (a). As a result of austenitising, the microstructure was transformed to FCC austenite phase. The rapid cooling resulted in carbon to diffuse into the martensite more than the saturated state. Hence, the excess carbon super-saturated martensite is formed [19]. In partitioning, initially excessive carbon present in the super saturated martensite starts to move out from martensite to retained austenite. So the microstructure developed after 15 sec of partitioning time will contain large amount of martensite as matrix with retained austenite in small fraction as illustrated in the Figure 2 (b). When the partitioning time was increased to 30 sec, fractions of retained austenite increased and that of martensite decreased as carbon diffused out from martensite to the retained austenite as shown in Figure 2 (c).

In case of further 60 sec partitioned sample again volume fraction of retained austenite was increased as shown in Figure 2 (d). At this stage fine epsilon carbide formation occurs at interfaces of martensite and retained austenite [20-21]. Under light optical microscope (LOM), epsilon carbide are not visible in microstructure but fraction of retained austenite can be observed.

For partitioning time of 120 sec, fine epsilon carbides might grow and coarsen due to partial decomposition of retained austenite and volume fraction of retained austenite is slightly decreased as visible in Figure 2(e).



**Figure 2** : Micrographs of AISI-4340 low alloy steel at 500X for partitioning time; (a) Without heat-treatment ( 0 sec) (b) 15 sec (c) 30 sec (d) 60 sec (e) 120 sec (f) 180 sec.

With increase in partitioning time up to 180 sec the volume fractions of retained austenite slightly decreased as revealed in Figure 2 (f). As epsilon carbide is metastable carbide, so for partitioning time of 180 sec unstable epsilon carbides are transformed into stable cementite,  $Fe_3C$ .

### 3.2 Hardness Testing

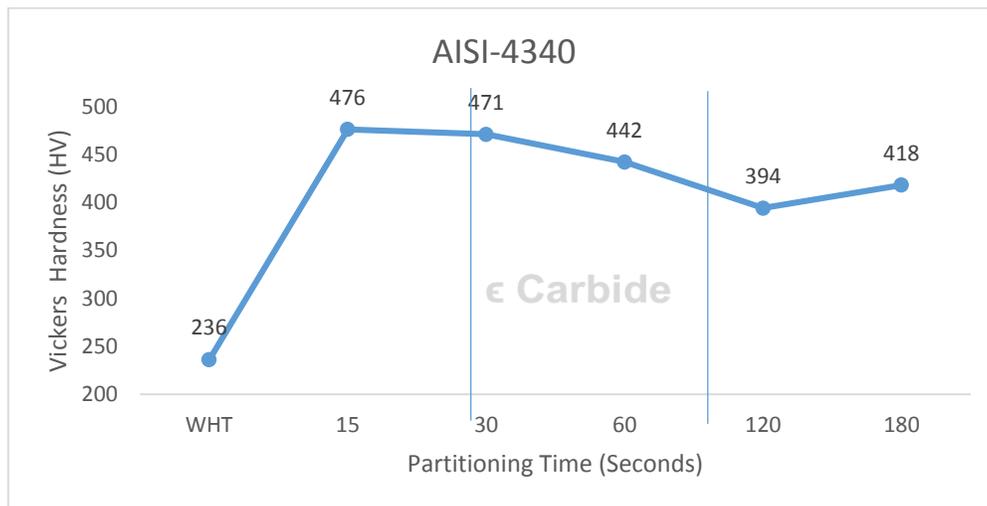
Vickers Hardness values of quenched and partitioned AISI 4340 steels are given in Table 4.

**Table 4: Vickers Hardness values of AISI-4340**

Sample ID	WHT	Partitioning Time ( sec)				
		15	30	60	120	180
Hardnes	236	47	471	442	394	418

The hardness values were strongly affected by Q and P heat treatment process. It is an established phenomenon that quenching increases the hardness of steel due to presence of martensite phase. The hardness values given in Table 4 are plotted as graph as shown in Figure 3. Partitioning time also affected the hardness values and a non-linear trend was observed as revealed in Figure 3. The Vickers hardness of non-heat treated or as received sample was 236 HV. But the hardness of the quenched and partitioned sample with 15 sec partitioning time was 476 HV. This increment in the hardness was due to the formation of high volume fraction of martensitic because of quenching and partitioning process. As discussed in section 3.1 about the microstructural changes, during the partitioning step excessive carbon of super saturated martensite diffused into retained austenite. So from 15 sec onwards, with a slight increase of partitioning time, volume fraction of martensite was decreased with increase in volume fraction of retained austenite phase. Resulting in drop in hardness to 471 HV after partitioning of 30 sec due to decrease in volume fraction of martensite which became evident from the microstructures of 15 and 30 sec partitioned samples as revealed in Figures 2 (b) and 2 (c) respectively.

Up to 30 sec partitioning time, excessive carbon was transferring from super saturated martensite to retained austenite but as the partitioning time was increased, the hardness was dropped from 471 to 442 HV for the sample partitioned for 60 sec as shown in Figure 3. The hardness of quenched and partitioned steel samples still remains higher than that of non-heat treated sample (236 HV), could be due to the formation of carbides and martensitic microstructure.



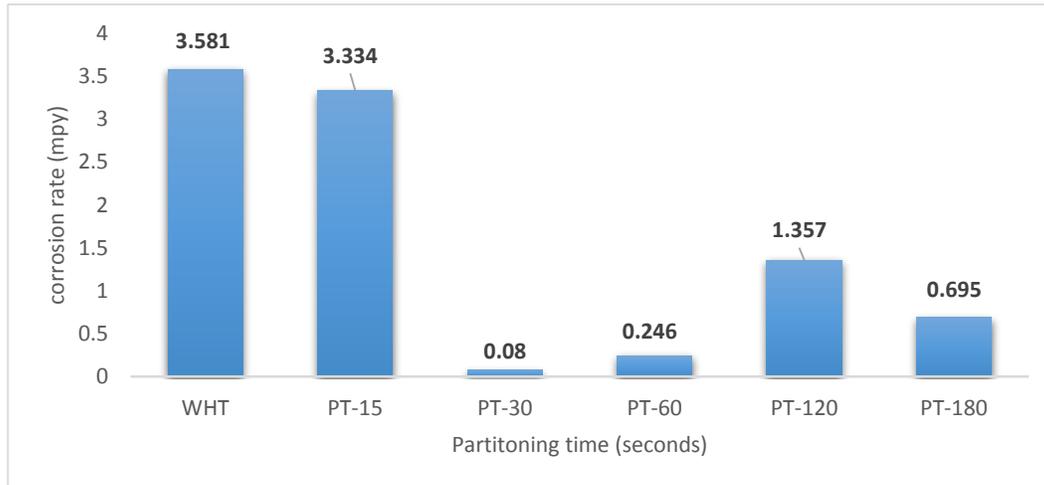
**Figure 3: Graph showing Vickers hardness vs. partitioning time of AISI 4340 Steel**

At this partitioning time of 60 sec, carbon moved from martensite and retained austenite could form epsilon carbide. Further increase in partitioning time up to 120 sec fine epsilon carbides were grown and coarsen resulting in further decrease in hardness to 394 HV. Since, epsilon carbide is unstable phase, any further increase in partitioning time would transform it into cementite  $Fe_3C$ . Due to formation of cementite, hardness was increased to 418 HV for 180 sec partitioning time.

### 3.3 Electrochemical Testing

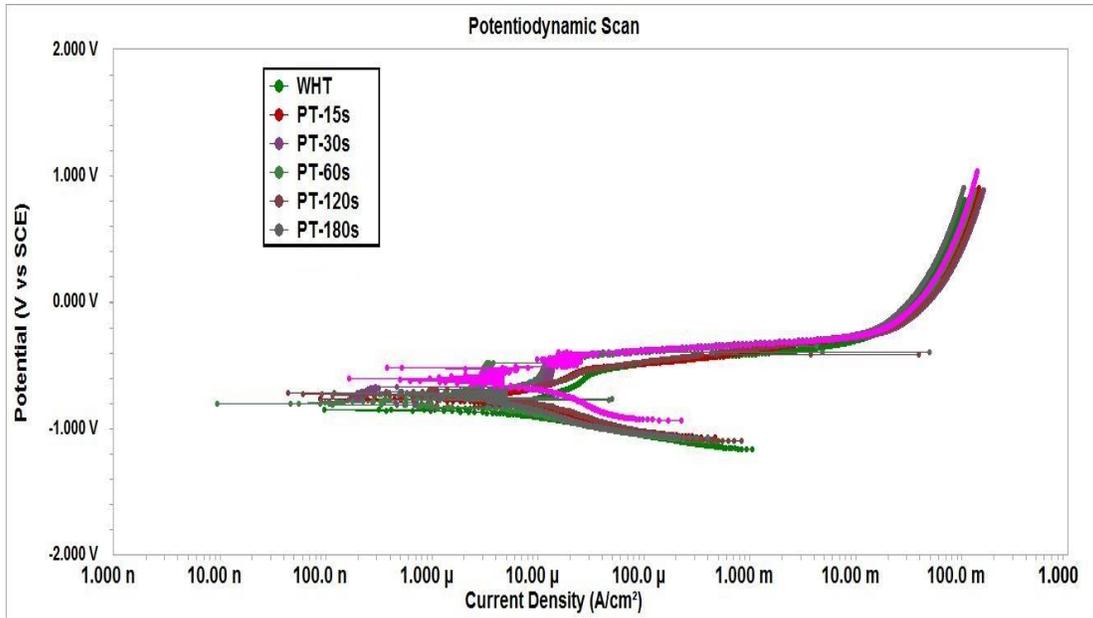
Non-heat treated sample of AISI-4340 steel exhibited higher corrosion rate than Q & P heat treated samples. The bar chart given in Figure 4 shows comparative corrosion rates of steel samples quenched and partitioned at different partitioning time periods. After doing Q and P heat treatment process, corrosion resistance was improved considerably as revealed in Figure 4.

The sample partitioned for 15 sec contained large volume fraction of martensite phase with retained austenite indicating high corrosion rate could be due to high carbon martensite as an active phase. Corrosion rate was rapidly dropped for the sample partitioned for 30 seconds.



**Figure 4: A comparison of corrosion rate of different samples as a function of Partitioning time of AISI 4340 Steel**

This change could be due to decrease in carbon content of martensite and increase in volume fraction of retained austenite. However the corrosion rate was slightly increased with partitioning time from 60 to 120 sec. This slight increase could be due to formation of epsilon carbides. Moreover, the corrosion rate again decreased with an increase in partitioning time from 120 to 180 sec as shown in Figure 4. This decrease in corrosion rate could be due to transformation of epsilon carbides into cementite.



**Figure 5: Comparison of Potentio-dynamic scans of different Q and P heat treated samples of AISI 4340 Steel**

Figure 5 presented Anodic Polarization Curves of studied samples. From the graphs the values of  $I_{corr}$  and other kinetic parameters were computed and are given in Table 5.

**Table 5: Kinetics Parameters of Potentio-dynamic Scan of AISI-4340 Q&P heat treated steel samples**

Sample No	Sample ID	$I_{corr}$ ( $\mu A/cm^2$ )	$E_{corr}$ (mV)	Corrosion rate (mpy)
1	WHT	7.830	-853.0	3.581
2	15	7.290	-767.0	3.334
3	30	0.176	-731.0	0.080
4	60	0.538	-802.0	0.246
5	120	2.970	-727.0	1.357
6	180	1.520	-720.0	0.695

$I_{corr}$  values showed the maximum corrosion current density  $7.830 \mu A/cm^2$  for non-heat treated sample and a minimum current density of  $0.176 \mu A/cm^2$  for the sample partitioned for 30 sec.

#### 4. Conclusions

From the present study the following conclusions could be drawn:

- After doing Q and P heat treatment process the volume fraction of the retained austenite was first increased and then decreased with an increase in partitioning time.

- The Vickers hardness of steel was increased from 236 to 477 HV after performing Q & P treatment and was decreased slowly with increase in partitioning time.
- The Corrosion rate of 3.581 mpy for the non-heat treated sample was considerably decreased to 0.08 mpy for the time period of 30-second after doing Q and P heat treatment.

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