

# Formation of nanocrystalline surface for AISI 304 stainless steel and DIN ck15 carbon steel and its effect on corrosion and Wear corrosion resistance.

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## Abstract

In this study, two essential goals were followed: 1) formation of Nano-crystallization of surface by shotpeening, 2) Evaluation of wear corrosion characteristics in solution with pH =2. These works were done on 304 stainless steel and ck15 low carbon steel. Nano-crystallization layer surface on steels were formed by severe plastic deformation done by shot peening. Formation of Nano-crystallization was checked by XRD. The layer produced on steel surface had a significant hardness containing residual compressive strength which not only improved fatigue life by 20% but also increased the wear corrosion resistance of the samples. The evaluation of wear corrosion was conducted in several pH and 1% NaCl solutions with two different load pressures, the trend of wear corrosion was examined by weight loss and DC polarization tests.

**Key words:** wear corrosion, corrosion, polarization, weight loss, Nano-crystallization.

## Introduction

Recently many researchers have paid more attention to Nano-crystalline surfaces of materials since it has been proved that Nano-crystalline surface may improve fatigue and friction resistance of materials [1-4]. Many investigators worked on the wear and friction behavior of low carbon steel with Nanocrystalline surfaces in dry condition and found a better wear and friction resistance [2, 3]. Shyrokov et al [4] evaluated wear corrosion behavior for surfaces in various acidic solutions by measuring the open circuit potential for formation of film using Pourbaix diagrams.

Azar et al [1] reported that shot peening can improve corrosion and fatigue resistance of 316L stainless steel. They also showed that by increasing the time of shot peening process the sample will be able to produce more passive film during wear corrosion tests [1].

The goal of this research was to evaluate the wear corrosion and corrosion resistance of two types of steel which their surfaces had Nano crystalline structures in solutions of 1% NaCl and acid solution with pH=2. The Nano-crystalline surfaces were produced by shot peening process.

## Materials and Methods

### Design of wear corrosion device

At the first step a machine was designed for rotational wear evaluation in corrosive electrolyte. This design was based on ASTM –STP 1000. All parts exposed to corrosive electrolyte were made of PE (poly ethylene) or PP (poly propylene). This machine had a rotational shaft which can transmit the weight load (up to 10 kg) to the sample during rotation. The machined had an inverter which can control the rotational speed (6 to 300 rpm). Electrolyte cell was designed to have a cap which could hold the reference and counter electrodes, the working electrode connection was placed at the bottom of the cell which could easily connect to a Potentiostat instrument [3],[19–20]. Figure 1 shows the complete design.



Figure 1– rotational wear corrosion machine

### Sample preparation and necessities :

All steel samples were prepared and machined to fit in sample holder according to wear corrosion machine. Then their surface rubbed by abrasive grits up to 1000. Grain size for CK15 was approximately 40  $\mu\text{m}$  and for the 304 stainless steel was 20 to 150  $\mu\text{m}$ .

Identical samples were also shot-peened according to SAE j442, 3 and other references [1–7] and [11–18]. Shot shapes were spherical, their diameters were 0.85 mm and made according to SAE j827. The shot peened machine was calibrated by Almen strip Type "A". The saturation point of the machine was approximately 1 mm in 60 second. To have 100% Nano-crystalline surface coverage on the samples, the number of passes which the samples should go through the shot-peening machine was calculated by the following equation according to SAE j2277 and reference [6]:

$$C_n = 1 - (1 - C_1)^n \quad (1)$$

$C_1$  = percentage of coverage in the first pass,  $C_n$  = 98% (100% actually is impossible),

$n$  = the number of passes.

$$n = \frac{\log(0.02)}{\log(0.7)} = 10.9 \sim 11 \text{ for 304}$$

$$n = \frac{\log(0.02)}{\log(0.6)} = 7.65 \sim 8 \text{ for ck15}$$

Each pass = 20 second (for experiment machine)

After shot-peening process, XRD was done on each sample to identify the grain size for steel [3, 9–10, 21–22]. For each type of steel two samples were selected as one with original surface and one with shot-peened surface. Because of strain produced on the sample by shot-peening process, a correction should be taken into consideration when using scherrer equation (2).

$D = \frac{K\lambda}{B \cos\theta}$  where  $K$  is constant and can vary between 0.87 to 1 and usually is 1.  $B$  is calculated as below:

$B_i$  is the fwhm for polished sample and  $B_o$  is the fwhm for shotpeened samples.  $\cos\theta$  can be calculated by each bragg angle. Unit of  $B$  is radian and unit of  $\theta$  is degree.

Parameter B for instrumental broadening should be changed to  $B_{\text{strain}} = B_0 - B_i$  (for Lorentzian patterns) [3]. the strain can be calculated by hall -Williamson relation. [9] The identifications of XRD instrument were:

Model: 303-PTS-Seifert , Current = 30 m A , tube voltage = 40Kv ,  
Anode Material = Cu wave Length  $\lambda_1 = 1.54059 \text{ \AA}$  , wave length  $\lambda_2 = 1.5442 \text{ \AA}$  ,  
Scan rate =  $4^\circ/\text{min}$  Scan from 30 to 140 degree

The average roughness of shot-peened samples was measured by an automatic device named "TR210" made by TIME GROUPE INC. With accuracy  $\pm 1.5 \mu$

The hardness of the surface was measured by "Straus Micro Hardness device".

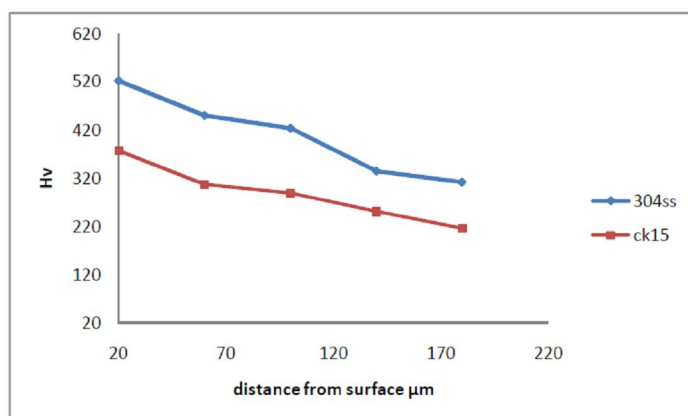
## Result and discussion

### Samples roughness

After shot-peening process the average roughness was:  $R_a$  for CK15 =  $7.36 \mu\text{m}$  ,  $R_a$  for 304stainless steel =  $4.47 \mu\text{m}$  which means adhesive wear depends on the average roughness [8] therefore, the adhesive wear of 304stainless steel is less than CK15 low carbon steel.

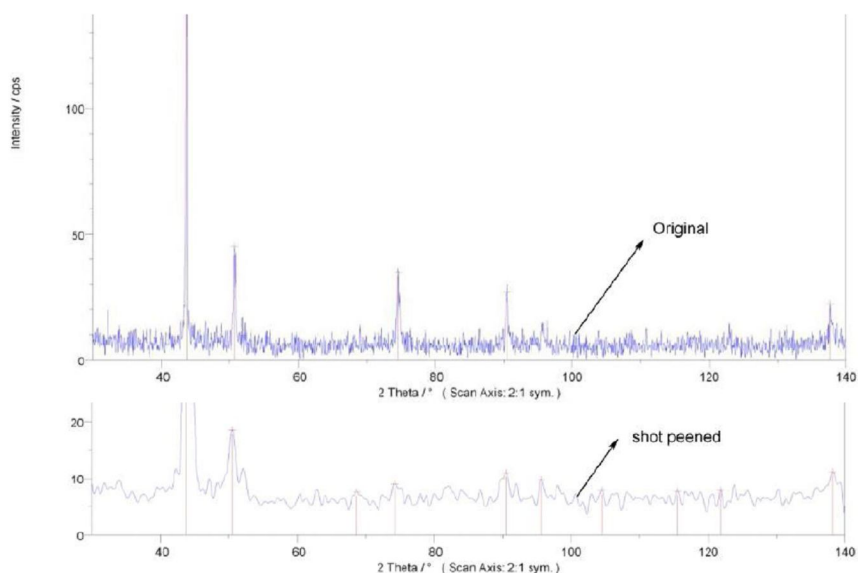
### Microhardness

Figure 3 shows the Micro hardness for the samples after shot-peening process as a function of distance from the surface. The surface hardness for 304 stainless steel was 533 HV and for CK15 low carbon steel was 377 HV. The micro-hardness from the surface to deeper regions of samples shows that the Nano-size grain is more than the deeper parts. This was occurred for both types of samples. ( the test were carried out by 50 gr. weight and diamond indenter and softer phases were selected)



**Figure 2– Micro hardness as a function of hardness for the Nano sized grain samples of 304stainless steel and CK15 carbon steel.**

Figure 3 is XRD plot for the Nano–sized grain surface for 304 stainless steel.



**Figure 3– XRD pattern for Nano sized grain surface of 304 stainless steel sample**

Parameters computation were done from data obtained from XRD plots and according to references [3,9] and listed in Tables 1 and 2.

Table 1 –parameters extracted from XRD plots for shot-peened304 stainless steel.

Brag Angel ( $2\theta^\circ$ )	43.6708	50.6683
FWHM(rad)original	0.2484	0.4254
" shot peened	0.6073	1.1871
D(meter)	$26.49 \times 10^{-9}$	$12.87 \times 10^{-9}$
Dm = $19.68 \times 10^{-9}$ (meter) average grain size		

Figure 4 is XRD plot for Nano-sized grain surface CK15 carbon steel.

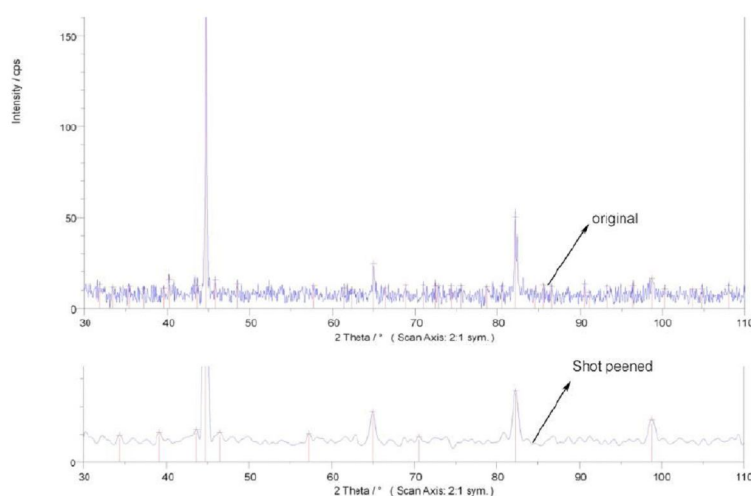


Figure 4– XRD pattern for CK15 carbon steel

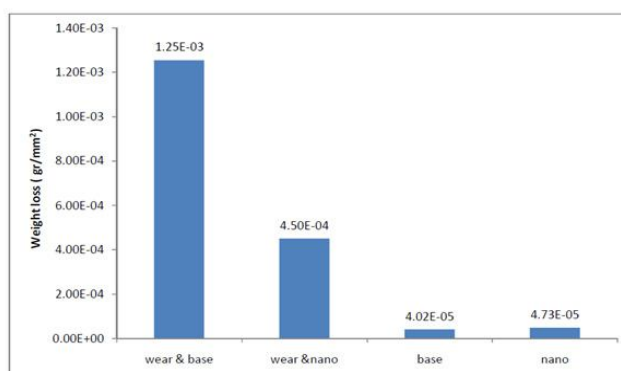
Table 2– parameters extracted from XRD plots for shot-peened CK15 carbon steel. Dm is an average grain size.

Brag Angel ( $2\theta^\circ$ )	44.7256	64.9827	82.155	98.7027
FWHM(rad)original	0.2354	0.3749	0.4547	0.4573
" shot peened	0.3227	0.5103	0.5617	1.0201
D(meter)	$109 \times 10^{-9}$	$77.3 \times 10^{-9}$	$109.510 \times 10^{-9}$	$240 \times 10^{-9}$
Dm = $80 \times 10^{-9}$ (meter) average grain size.				

Based on the data presented in Tables 1 and 2, the size of grains at the surface of the samples were of Nano sized one.

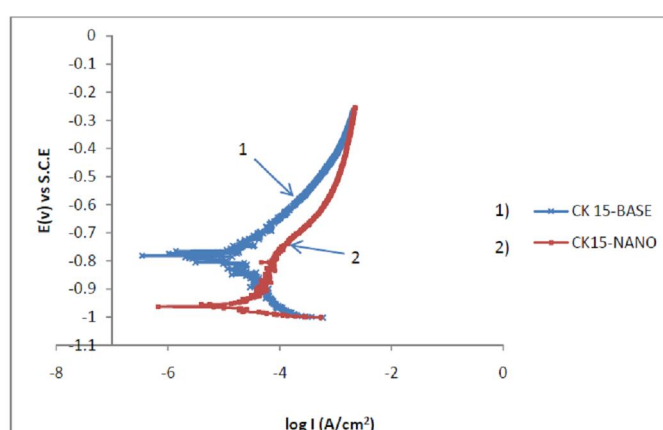
Figure 5 is the result of weight loss test for the sample of CK15 steel in 1% NaCl solution with pH=2 for different condition of wear.





**Figure 5– weight loss for the sample of CK15 steel in 1% NaCl solution with pH=2 for different condition of wear. Measurements were made after 10 hours.**

The results on Figure 5 show that corrosion rate of steel for shot-peened surface and without that is almost the same. However when these samples are under wear corrosion, the rate of corrosion for sample with no shot-peening is very high in comparison to that of the sample with shot-peened surface. Figure 6 is a plot of cyclic polarization for the CK15 steel in 1% NaCl solution with pH=2 for shot-peened surface and without that. This figure shows that the uniform corrosion for the sample with shot-peened surface is higher than that without shot-peening in any voltage. However, the samples with shot-peened surface show a film on its area. The sample without shot-peened surface shows no film on its surface.



**Figure 6 is a plot of cyclic polarization for the CK15 steel in 1% NaCl solution with pH=2 for shot-peened surface and without that.**

Figure 7 is weight loss plot for the sample 304 stainless steel with and without shot-peened surface in 1% NaCl solution with pH=2 after 10 hours. This figure shows that corrosion of 304 stainless steel is very small for surfaces with and without shot-peening. However, when samples exposed to wear corrosion, the sample without shot peening has higher rate of corrosion in comparison to that with shot-peened surface. This may be due to the fact that film on the Nano-grain size surface is more stable than that of the surface without Nano-grain size. This can clearly be seen in Figure 8 which is a cyclic polarization for the sample with and without Nano-sized grains. But the main reason due to comparison of both without and with shot peening samples is hardness of surface that increased by shot peening.

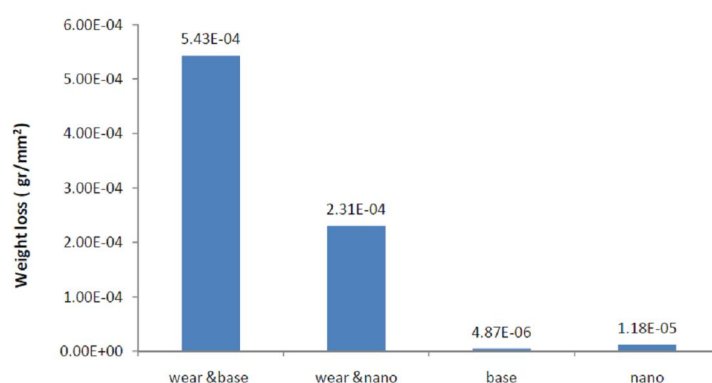


Figure 7– is weight loss plot for the sample 304 stainless steel with and without shot-peened surface in 1% NaCl solution with pH=2 after 10 hours.

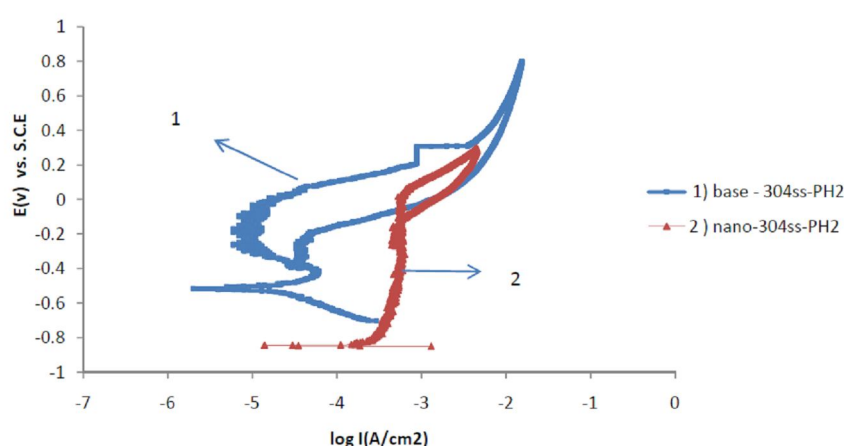


Figure8–cyclic polarization curves for 304 stainless steel in solution with 1% NaCl and pH=2 with and without shot-peening.



## Conclusions

- 1- The grain size of shot-peened surface was in range of Nano-size scale.
- 2- Uniform corrosion rates for both samples of CK15 carbon steel and 304 stainless steel for Nano-sized grain surfaces were higher than that of ordinary surfaces in 1% NaCl solution with pH=2.
- 3- Surfaces prepared with shot-peening process for both samples of CK15 carbon steel and 304 stainless steel under wear corrosion in 1% NaCl solution with pH=2, showed excellent resistance in comparison to that without shot-peening process. The main reason of Wear corrosion resistance is due to difference of increased hardness that is created by shot peening on the surfaces of samples.

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