A study on the surface severe plastic deformation and corrosion behavior of an interstitial free steel

Hamed Eskandari1, Mohsen Saboktakin Rizi2, Arezoo Ghanbari1, Babak Nasiri3 and Kamran Dehghani1

1 Department of Materials and Metallurgical Engineering, Amirkabir University of Technology, PO Box 1873-4413, Tehran, Iran
2 Department of Industrial Engineering, Lenjan Branch, Islamic Azad University, Isfahan, Iran
3 Basser Kala Co., Tehran, Iran
E-mail: hamed_62_esi@yahoo.com

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Abstract
In the present work, a Ni-stabilized interstitial free (IF) steel was subjected to severe plastic deformation (SPD) in order to attain nanograin structure. To do this, a different technique so-called brushing was used. This technique is very cost-effective, simple and practical. For brushing process, different brushes made of brass, stainless steel and low carbon steel were used. The processing parameters such as brush rotational speed, different shapes and dimensions of the brush wire, the amount of brushing and applied load were adjusted so that to attain the desirable structure. The surface and microstructural evolutions were characterized by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM). The thickness of the nanostructure surface layer formed by brushing was about 40–50 μm with a maximum microhardness of about 540 Vickers. The formed nanostructure layer had a grain size of 30–50 nm. In order to study the effect of nanograin structure on the corrosion behavior, the samples, brushed under different conditions, were subjected to corrosion testing and the related polarization curves were obtained using 3% NaCl solution. The comparison of the polarization curves of brushed and non-brushed samples indicated that the peak of polarization curve of brushed sample, exhibited a larger potential difference and a lower current density comparing to not-brushed case. The larger potential difference and the lower current density mean greater corrosion resistance and lower corrosion rate. A quantitative comparison between the current density of the two samples revealed a 55% increase in corrosion resistance for the brushed sample as compared to not-brushed counterpart.

1. Introduction
Interstitial free (IF) steels have a ferritic single-phase microstructure and are among the ultra-low steels. Besides, because of the presence of micro-alloy elements such as titanium and niobium, all the amounts of carbon and nitrogen are tied up leading to the so-called IF steels [1, 2]. However, their yield strength is very low due to the reduced amount of interstitial atoms, i.e. carbon and nitrogen and in turn the low volume fractions of carbides and nitrides. The latter can significantly promote the grain growth [3, 4] as the grain mobility is enhanced due to diminished effect of Zener pinning. This also leads to a low strength according to the Hall-Petch equation. Thus, the problem with the IF steels is their low yield stress. On the other hand, SPD is the promising approach to reduce grain size from micro to nano scale.

It is possible to harden or increase strength in various ways. However, the most hardening mechanisms reduce ductility and formability [5]. Among different mechanisms, decreasing the grain size is the only mechanism by which the strength is increased while ductility is not changed [6]. The grain size of the polycrystalline material plays an important role in determining many major properties such as strength and resistance to plastic flow. In general, fine-grained materials have many advantages over coarse-grained materials. Nanostructured materials are polycrystalline materials with grain size on the order of a few nanometers [7].
Today, nanostructured materials have attracted a great deal of attention due to their unique properties. Nanostructured materials have a large volume fraction of grain boundary, resulting in significantly different physical, mechanical, and chemical properties as compared to coarse-grained polycrystalline materials. Nanostructured materials exhibit higher strength and hardness, higher toughness, higher permeability, larger specific heat capacity, larger thermal expansion coefficient, and excellent magnetic properties when compared to conventional polycrystalline materials [8]. To the present day, various methods have been developed to fabricate such materials. One of the main methods for fabricating nanostructured materials is severe plastic deformation. Application of severe plastic deformation for producing bulk nanocrystalline materials has attracted wide attention among researchers. At present, researchers mostly focus on the mechanisms for reducing grain size and grain boundary structure caused by severe plastic deformation. Recently, it has been shown that severe plastic deformation can lead to unique phenomena such as unusual plastic flow behavior during torsion, amorphization in two-phase alloys, non-equilibrium diffusion, the formation of super-saturated solutions, and the dissolution of sediments in alloys [9]. So far, the severe plastic deformation processes have been studied on non-ferrous metals and alloys, and a limited number of studies were reported to have focused on the effect of severe plastic deformation on ferrous metals. Verma et al. achieved a grain size of 300 nm through equal-channel angular pressing and annealing processes of an interstitial free steel at a temperature of 600 °C–700 °C [10]. Jindal et al. studied the effect of accumulative roll bonding on the microstructure and mechanical properties of IF steel at a temperature of 950 °C. They achieved a grain size of 22 μm and the hardness of 200 Vickers in four accumulative roll bonding passes [11]. Cizek et al. achieved a grain size of 580 nm in IF steel through high-pressure torsion [12]. Saray et al. examined the formability of ultrafine-grained (UFG) IF steel and achieved a grain size of 240–510 nm through accumulative roll bonding [13]. Tsuji et al. achieved a grain size of 420 nm in IF steels through accumulative roll bonding at temperatures above 773 °K [14]. Tamimi et al. achieved grain sizes around 300 nm through accumulative roll bonding in IF steel at 823 °K [15]. Although many researchers have aimed to achieve grain sizes below 100 nm in IF steel, their findings indicate that the minimum grain size obtained through severe plastic deformation processes was only around 300 nm. Stacking-fault energy (SFE) levels play a key role in determining the final grain size of materials subject to accumulative roll bonding. Since SFE in IF steel is about 200 mJ m−2, restoration can easily occur in this type of steel and prevent the accumulation of dislocations and the occurrence of recrystallization [16–21]. The present study aimed to investigate the possibility of achieving a nanostructure on the surface of IF steel by wire brushing and analyzing its corrosion properties.

2. Experimental procedure

The composition of studied IF steel is listed in table 1. Figure 1 shows the initial microstructure having an average grain size of about 50 μm.

| C  | Si   | Mn   | Cr   | Ni   | P   | S   | Mo | Fe |
|----|------|------|------|------|-----|-----|----|----|
| 0.002 | 0.0054 | 0.0744 | 0.0103 | 0.0116 | 0.0135 | 0.005 | 0.0052 | Bal. |

Wire brushing was employed as a SPD technique to create a nanostructure layer on the surface of studied IF steel. In order to examine the different testing conditions such as brush rotational speed, different shapes and dimensions of the brush wire, the amount of load on the surface of the sample, working temperature and brushing time, a brushing machine was designed and fabricated (figure 2).

In order to investigate the effect of different brushes on the microstructure and mechanical properties, the tests carried out using a variety of brushes made of carbon steel, stainless steel, and brass with different dimensions and specifications as reported in table 2.

To study the effect of different conditions on the thickness of the nanostructured layer and also the resulting hardness, the tests were carried out under different conditions (tables 3 and 4) on IF steel samples.

The microstructural evolution of the brushed samples was studied by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM). For TEM studies, the samples were polished up to a thickness of 100 μm using the sandpaper (having the grit sizes 60–2200) along with the 0.05 μm alumina powder. This was followed by jet polishing using an electrolytic solution (acetic acid 93% and perchloric acid 7%). To measure in-depth hardness from 0–160 μm, hardness test was carried out using HVS-1000 micro-hardness tester. The 100 g load and 20 s indentation were applied. X-ray diffraction was used to identify the existing phases (Philips X’Pert-MPD, 40 kV, 30 mA). Cu-Kα radiation with a wavelength of 1.5404 Å was used in all the tests. The grain size of nanostructure surface...
layer was determined using Williamson-Hall equation [20].

\[ \beta_{(h\bar{h}k)} \cos \theta = \frac{K \lambda}{D} + 4 \varepsilon \sin \theta \]

Where \( D \) is the grain size and \( K \) is a coefficient ranging from 0.89 to 1.39 according to the size and shape of the grain. \( \lambda \) is the wavelength of x-ray, \( \theta \) is the angle, and \( \beta \) is the peak width at half-maximum height. In order to study the effect of nanograin structure on the corrosion behavior, the samples brushed under different conditions were subjected to corrosion testing and the related polarization curves were obtained. The corrosion behavior of the samples was examined in a 3% NaCl solution using AUTOLAB system.

3. Results and discussion

3.1. Hardness measurements

After the brushing process under different conditions, microhardness test was performed through the thickness to estimate the depth of the nanostructure layer. Figure 3 depicts the microhardness profile obtained from the surface toward the inner sections of the different samples. Accordingly, the sample tested under the testing condition No. 5 exhibits greater hardened depth, while the sample tested under condition No. 2 exhibits the maximum surface hardness. Other samples exhibit almost the same trend. Generally, the maximum microhardness (550 Vickers) was achieved on the surface of the sample under condition No. 2 whilst the maximum thickness of the nanostructure layer, which was about 50–60 \( \mu \)m, was pertaining to condition no. 5. It is also observed that due to the brushing process, the surface microhardness was also increased by about 3 to 4 times that of the untreated sample. According to Hall-Petch equation, there is a direct relation between the strength/hardness and grain size. Therefore, such an increase in hardness can be attributed to the nanograin formed by brushing.

Since both the hardness and uniformity of the layer formed during brushing are important factors in determining the optimal conditions, the microstructure of the samples was also examined.
The images in figure 4 show the penetration depth after harness testing of samples brushed with brush No. 1 under the conditions listed in table 3. As illustrated, the thickness of the microstructure surface layer formed by brushing is about 40–50 μm with a maximum microhardness of about 540 Vickers.

Table 2. Specifications of all the brushes used.

| Brush no. | Wire material | Wire length (mm) | Wire diameter (mm) | Total number of wires | Brush diameter (mm) | Brush thickness (mm) |
|-----------|---------------|------------------|-------------------|-----------------------|---------------------|----------------------|
| 1         | S.S           | 20               | 0.3               | 2500                  | 70                  | 10                   |
| 2         | Brass         | 26               | 0.15              | 12000                 | 100                 | 20                   |
| 3         | Steel         | 16               | 0.3               | 15500                 | 105                 | 30                   |
| 4         | S.S           | 23               | 0.3               | 5500                  | 100                 | 18                   |
| 5         | Steel         | 18               | 0.3               | 1500                  | 60                  | 18                   |
| 6         | Steel         | 13               | 0.15              | 8000                  | 64                  | 10                   |

Table 3. Differing testing conditions to study the thickness and hardness of the nanostructured layer.

| Test no. | Brush no. | Brush rotational speed (rpm) | Load (Kg) | Brush linear speed (m s⁻¹) | Brushing time (s) | Description |
|----------|-----------|-------------------------------|-----------|-----------------------------|-------------------|-------------|
| 1        | 1         | 27000                         | 2.2       | 83.7                        | 60                | —           |
| 2        | 1         | 20000                         | 1.7       | 83.7                        | 60                | —           |
| 3        | 1         | 27000                         | 1.7       | 113                         | 60                | —           |
| 4        | 1         | 27000                         | 2.2       | 113                         | 10                | —           |
| 5        | 1         | 20000                         | 2         | 83.7                        | 5                 | —           |
| 6        | —         | —                             | —         | —                           | —                 | Control sample |
Table 4. Different conditions of the second stage of tests with different brushes.

| Test no. | Brush no. | Brush rotational speed (rpm) | Load (Kg) | Brushing time (s) |
|----------|-----------|------------------------------|-----------|-------------------|
| 7        | 2         | 20000                        | 3.5       | 60                |
| 8        | 3         | 20000                        | 2.5       | 30                |
| 9        | 3         | 20000                        | 1.5       | 30                |
| 10       | 4         | 12000                        | 1         | 60                |

Figure 3. The effect of different testing conditions on the thickness and hardness of the deformed layer in IF steel.

Figure 4. (a) Optical microscope images of the microstructure layer and the penetration depth in the sample brushed under the conditions listed in table 3, (b) the microhardness curve versus distance from the surface.
3.2. Surface and microstructural evolutions

Figure 5 shows the optical microscope images of the surface of samples brushed under different brushing conditions.

The images in Figure 5 taken from different regions of surface indicate the severe plastic deformation employed by brushing. According to this figure, three regions can be recognized. The first and the top region is a severely deformed layer. The bottom region, which is located beneath the first region, has been affected by the deformation force and the grains were elongated. Finally, the third region is pertaining to the base material.

Figure 5(a) shows the image of sample No. 1, according to which the level of deformation due to the brushing process in this sample is low and negligible in the first region and the surface layers. The applied load resulted in a slight deformation in the second layer. In figure 5(b), the applied load was reduced and the process time was increased compared to sample No. 1. In this sample, the first layer and the severely deformed regions are separated. The second region and sub-surface layer include a small region. For the case No. 1, the motor speed was 700 rpm higher than that in the case No. 2. Sample No. 3 surface exhibits both a severely deformed structure and a good uniformity between the initial layers. In general, the microstructural evolutions reveal the occurrence of severe plastic deformation.
of severe deformation in the workpieces subjected to brushing; though, different thicknesses experience different strains. As severe deformation requires employing larger loads, the brushes made of stainless steel and brass were used for further examination (Table 4).

In case of test No. 7 carried out with a brass brush, it was tried to increase the parameters such as time, force and speed as high as possible. Tests No. 8 and 9 were performed with a steel brush, which had greater strength than brush No. 2 and in turn required smaller force and shorter time. In test No. 10, a stainless steel (brush No. 4) was used. However due to its diameter, width, and compactness, the force and motor speed were decreased as much as possible to avoid any damage to the sample during brushing. Since wire of brushes No. 2, 3, 5, and 6 are made of brass and carbon steel, they have lower or similar strength as studied IF steel. Consequently, they had no significant effect on the samples even after changing the mentioned parameters. Therefore, the stainless-steel brush was used for other tests. Comparing the brushes No. 1 and 4, the former exhibited better results owing to its smaller diameter and width. Hence, the optimal conditions listed in Table 5 were applied for testing in order to create a nanostructure surface layer in the studied IF steel.

An important aspect to consider is the formation of a rough surface after brushing. Figure 6 is an optical microscope image of the brushed surface of the present IF steel. For better comparison, the surface topography was determined before and after brushing. Figure 7 shows the surface topography of the sample before brushing. In this case, the initial surface roughness (Ra) was about 0.42 μm, whereas the topography after brushing indicates the Ra of about 4.25 μm. Therefore, the surface roughness has been increased by brushing.

As previously mentioned, the sample is composed of three regions after brushing. The top layer having the maximum hardness is well distinguishable from the second region just beneath the surface. Comparing to the top surface or first region, the second region experiences less deformation having elongated grains. Finally, the third region as the inner layer exhibited no deformation as the initial structure. Although there is a very clear boundary between the first and second region, the transition from the second to the third region is a gradual transition. In addition to the optical microscope images, the SEM technique was also used to characterize the second region. Figure 8 shows the SEM image taken from the second region indicating the formation of UFGs.

The TEM method was used to further characterize the nanostructure layer formed on the sample surface. As shown in Figure 9, the structure obtained after brushing includes a complex structure of dislocations. In high-SFE materials such as IF steels, the dislocations are rearranged into a three-dimensional cell structure after the deformation. The cell walls then become intertwined dislocations. The cell size depends on strain and material. According to the research works that focused on creating a nanoscale structure through severe plastic deformation, a restoration process is needed to rearrange the resulting dislocations and microstructures. The cell walls of dislocations become more thickened; some of the cell dislocations are eliminated, and the cells become subgrains through annealing. Rearrangement of dislocations in thickened walls, change the cell structure into subgrain boundaries. These microstructural evolutions can be considered as crucial stages during the restoration process. The samples were thus subjected to annealing for 30 min at 300 °C. According to the

| Brush no. | Brush rotational speed (rpm) | Load (Kg) | Brush linear speed (m s⁻¹) | Brushing time (s) |
|----------|------------------------------|-----------|-----------------------------|------------------|
| 7        | 20000                        | 1.7       | 83.7                        | 60               |

Figure 6. An optical microscope image of the IF steel surface brushed under conditions listed in Table 5.
Figure 7. Surface topography of the IF steel surface: (a) before brushing, (b) after brushing.

Figure 8. The SEM images of (a) Region 2, (b) Region 2–3 transition, and (c) Region 3.
images obtained from the annealed sample, the structure of the surface layer formed by brushing is well indicative of the nanostructure. As shown in the SAED pattern, after the annealing of the specimens, the ring pattern is less consistent, indicating that the grain has more distinct boundaries. The annealed sample consists of more clear subgrain boundaries with grain sizes ranging between 30 and 50 nm.

X-ray diffraction (XRD) was employed for phase identification in controls sample and brushesd sample under optimal conditions in table 5. The grain size of nanostructure surface layer was determined using Williamson-Hall equation. According to figure 10, the formed phases are ferrite with a grain size of around 30–50 nm.

In addition to OM, SEM and TEM studies, the atomic force microscopy (AFM) was also used to characterize the produced nanostructure (figure 11). The AFM images were obtained from the transverse section of the sample, i.e. from the top layer towards the mid-thickness of sample.

It is worth noting that the grain sizes obtained by AFM are slightly larger than those obtained by TEM. The reason can be that the TEM images are taken from the top layer where plastic deformation is the greatest leading to the finest/nanoscale grains. Whereas, the AFM images show the changes in the size of grains from the top surface towards the inside layers subjected to less deformation.

3.3. Corrosion behavior of nanograin layer
In order to study the effect of nanograin structure on the corrosion behavior, the samples brushed under different conditions were subjected to corrosion testing and the related polarization curves were obtained, figure 12. A comparison between the curves of sample 3 and sample 1 (untreated case) indicates that the peak of polarization curve of sample 3, i.e. the transition from the active region to the passive region, exhibits a larger

![Figure 9](image-url) The TEM image of the first (severely deformed) region (a) before annealing (b) after annealing.

![Figure 10](image-url) The XRD results for the (a) control, (b) brushed under optimal conditions and stress-relieved samples.
potential difference and a lower current density than sample 1. The larger potential difference and the lower current density mean greater corrosion resistance and lower corrosion rate.

The comparison between the current density of the brushed and untreated samples revealed a 55% increase in corrosion resistance for the former. Moreover, a comparison between the polarization curves of samples No. 2 and 3 shows a slight increase in potential difference and peak current density of sample No. 2 comparing to sample No. 3. Although, the brushing conditions for samples No. 2 and 3 were similar, sample 2 was resorted after brushing. Therefore, the occurrence of restoration phenomenon can lead to the formation of a passive layer consisting of Fe₃O₄ and γ-Fe₂O₃. According to figure 12 and comparing the curves of samples No. 3 and 4, it is obvious that the increase in brushing time from 30 to 60 s has no significant effect in terms of improving corrosion resistance. However, the force applied to the workpiece surface is reduced by changing the type of brush and using a brush with softer and finer wires. Consequently, the resulting structure consists of ultra-fine grains UFG rather than the nano-sized grains.

4. Conclusions

1. It was possible to achieve a nanostructure layer (grain size of 30–50 nm) on the surface of studied IF steel through brushing technique, which is a simple and cost-effective method for severe plastic deformation.
2. Three regions were recognized after brushing: (i) a nanostructure surface layer, (ii) an ultrafine-grained sub-surface layer, and (iii) the base metal.

3. The annealing after severe plastic deformation lead to the formation of distinct and clear grain boundaries.

4. The nano-structured IF steel showed about 50%–55% improvement in the corrosion resistance behavior comparing to untreated IF steel.

**ORCID iDs**

Hamed Eskandari  [https://orcid.org/0000-0002-5829-6065](https://orcid.org/0000-0002-5829-6065)

Mohsen Saboktakin Rizi  [https://orcid.org/0000-0003-1752-0005](https://orcid.org/0000-0003-1752-0005)

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