FRACTAL ANALYSIS OF THE PORES IN AISI316L SS IMPLANTED WITH OXYGEN AND HELIUM IONS

*Durowoju M.O, Onawumi A.S & Oladosu K.O.
Department of Mechanical Engineering, Ladoke Akintola University of Technology, Ogbomoso, Oyo State, Nigeria

ABSTRACT
Fractal analysis is used to numerically characterize the pores in AISI316L stainless steel to be able to study the shape, distribution and type of the pores. The fractal analysis uses two dimensionless parameters: fractal dimension $D$ and Sphericity, $\beta$. The AISI316L stainless steel is widely used in medical field as an implant material due to its good corrosion resistance and biocompatibility.

In this work AISI316L, stainless steel was implanted with two different ions: Oxygen and Helium separately at 100KeV with dose of $1 \times 10^{17}$ ions/cm$^2$ at room temperature. The crystallographic orientation and surface morphology were studied using x-ray diffraction analysis (XRD). The micro hardness was measured by Vickers method with varying load. The result indicated that the predominant pores in the virgin material are of spherical shapes with $\beta< 0.3$ and $D>1$. It was further observed that the pores are flake like type and clustered, thereby creating ease of linkage of the pores. The ion implantation showed a reduction in the pores, predominantly noodle-like types, (i.e. a shape change from spherical to round pores) and change in distribution from clustered to randomly spaced pores. The surface hardness is found to be 1202HV, 1020HV and 195HV for Helium, Oxygen and Virgin material respectively.

There is a significant improvement in the shape, type, and distribution of the pores in AISI316L stainless steel implanted with Oxygen and Helium. The Vickers hardness test shows that the micro hardness of AISI316L stainless steel implanted with Oxygen and Helium is higher than that of the virgin material.

Keywords: Fractal Analysis, Pores, AISI316L Stainless Steel, and Ion Implantation

1. INTRODUCTION
The use of fractals to study surfaces of different materials has been done by different researchers and it is still receiving increasing attention. Scientists who study or try to describe natural phenomena have to consider the use of fractal geometry. From the theory of chaos to land surface description, from sea surface synthesis to stock market analysis, fractal concepts are used in more and more research fields (Giuseppe et.al. 2006). Chung-Kung (1998) used the fractal analysis and observed the effect of heat treatment on the well measured nitrogen isotherms on alumina and aluminum borate samples. He observed that heat treatment, for the two methods used may decrease fractal dimension, $D$ of the four examined porous samples. From analysis carried out, fractured surfaces were discovered to be fractal in nature (Alexander, 1990).

For alloys and composite materials containing regular microstructures a prediction of mechanical properties can be made by a quantitative measurement of features such as grain size, particle size, and spacing etc. This however is not the case where an irregular microstructure is involved because of difficulty in a numerical characterization of the structure. For each microstructure the application fractal geometry offers a method by which both the individual particle shapes and the mode of distribution of the particles can be fully described in numerical manner (Shu-Zu and Hellawell,1994). Durowoju (2007) used the fractal analysis to observe the size, shape, and distribution of pores in Al-$V_2O_5$ alloyed composite. The analysis revealed that the pores are of irregular shapes, i.e Shrinkage pores, with $\beta< 0.3$.The graph of $\beta$ against $D$ shows that as $\beta$ decreases the $D$ values increases. Similarly, the sizes, shapes and distribution of the pores in samples of Al-20%wtMg heat-treated at 470$^0$C for a soaking time of 30 minutes was done (Durowoju et. al., 2009). The results show that in the graph of $\beta$ against $D$ there exist critical values of fractal dimensions (1.0360 and 1.0510) above which any increase in the fractal dimension causes a decrease in the sphericity. Similarly, there exists minimum values of fractal dimension and sphericity ($D= 1.048$ and $\beta= 0.0384$), above which the tensile strength and hardness increases. AISI 316L stainless steel is being widely used as biomaterials and materials of construction. More specifically, AISI 316L SS is used in the medical field as an implant material due to its unique property of good corrosion resistance and biocompatibility. The success of implant in the human body depends on many factors such as bio safety, biocompatibility and biofunctionality in the environment wherein the implant are placed. But if the body environment is harsh, this might result in corrosion of implants (Liu Chenglong et al.,2005).Although AISI 316L stainless steel shows extremely good corrosion resistance, it is nevertheless prone to defects (pitting, pores, voids e.t.c) . Surface modification techniques have been applied to improve the corrosion resistance of AISI 316L stainless steel (Fossati et al.,2006). The use of implantation as a surface modification method has been done by different researchers. Nitrogen was implanted in
Titanium, NiTi (Neonila Levintant – Zayonts and Stanislaw Kucharski, 2009). Implantation of helium also attracts considerable attention of researchers. Helium ions were implanted in polymers (Valenza et al., 2004). Also, Argon, nitrogen and oxygen were implanted in titanium modified SS316L (Kamachi Mudali et al., 1997).

In the present study, oxygen and Helium ions were implanted on AISI 316L stainless steel and in the microstructures, obtained pores were specifically looked at, and characterized using fractal analysis. The experiment was done following the procedure adopted by other researchers (Masaya Iwaki, 1999). This is still on going but the microstructures and hardness values obtained are used in the present study.

2. EXPERIMENTAL PROCEDURE

2.1. Sample Preparation and Ion Implementation

The AISI316L SS was used as the metal substrate and the elemental composition is given in Table 1. The AISI316L SS alloy in as-received mill annealed condition was cut into 8mm diameter and length of 5mm and 15mm size pieces. Prior to the study, the AISI 316L SS samples were polished using silicon carbide emery papers of 120, 220, 320, 400, 500, 600 and 800 grit. Final polishing was done using 5µm and 1µm diamond pastes in order to produce scratch-free mirror –finish surface. The polished specimens were examined using optical microscope for the presence of pits, pores or scratches on the surface. The polished specimens were washed with detergents solution, degreased with acetone and thoroughly washed with distilled water. They were further subjected to ultrasonic cleaning in acetone for 10mins. Finally the sample was rinsed in de ionized water, dried and used for further studies.

Table 1. Composition of the AISI 316 Stainless Steel (wt. %)

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>P</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L SS</td>
<td>0.020</td>
<td>1.10</td>
<td>0.469</td>
<td>16.49</td>
<td>10.02</td>
<td>2.01</td>
<td>0.015</td>
<td>0.013</td>
<td>Bal</td>
</tr>
</tbody>
</table>

The ion implantation of both the oxygen and helium ions was accomplished with a 150 keV accelerator. AISI316L SS had undergone an implementation of selected ions at an energy level of 100keV, dose $1 \times 10^{17}$ ions/cm$^2$ at room temperature. Ions were accelerated in a linear accelerator. This implantation process is a line-of-sight process.

2.2. Hardness Test

The hardness test was carried out to measure the micro hardness of both the virgin and the implanted specimens. The Ever One, Model no MH-3 (Germany) micro hardness testing machine was used to carry out the test. The hardness profile of the surface layers was measured with varying loads ranging from 10g to 100g. The Vickers hardness of AISI316L SS is found to be 195HV and it is in good agreement with the hardness values measured by the other authors (Chih-Neng Chang and Fan-Shiong Chen, 2003).

2.3. Fractal Analysis

Fractal geometry was developed (Mandelbrot, 1983) over two decades ago. Its principle is universal in any measurement and has been previously used to numerically describe complex microstructures including graphite flakes and nodules (Lu and Hellewell, 1994, 1995, 1999). The mathematical basis for measuring chaotic objects with the power law modified shall be adopted in this research. The basic equation is as follows:

$$P = P_e \delta^{D-1} \cdot \left(1 < D < 2 \cdot P \cdot \delta_m < \delta < \delta_M \right)$$

(1)

Where $P_e$ is the measured perimeter, $P$ is the true perimeter, $\delta$ is the yardstick, $\delta_m$ and $\delta_M$ are the upper and lower limits respectively, for any shape and $D$ is defined as the fractal dimension ($1 < D < 2$). From the above expressions it can be deduced that the true perimeter is actually a function of the yardstick for measurement. The smaller the yardstick used, the more accurate the measurement. This study intends to use the average of four different yardsticks for better accuracy.

The fractal dimension, $D$, therefore describes, the complexity of the contour of an object (Fig.1). It can be more practically called the roughness (Huang and Lu, 2002).

When $\delta < \delta_m$, the measurement is not sensitive to the yardstick chosen, therefore giving a smaller value of the slope, while $\delta > \delta_M$, the size of the yardstick exceeds that of the individual feature being measured so that the measurement loses meaning because the object falls below the resolution limit of the yardstick used for measurement (Lu and Hellewell, 1994).
Sphericity, $\beta$, another dimensionless number, is used together with roughness, $D$, to describe the shape of the pores formed. It can be expressed as

$$\beta = \frac{4\pi A_r}{P^2} \cdot (0 < \beta < 1 \cdot \text{and} \cdot 1 < D < 2)$$

(2)

substituting eq.(1) in (2) gives
\[ \beta = \left( \frac{4\pi A_T}{P_e} \right) \delta^{2(1-D)} \cdot \left( 0 < \beta < 1 \cdot \text{and} \cdot 1 < D < 2 \right) \] (3)

Where \( A_T \) is the total pore area. When \( \beta=1 \) and \( D=1 \), a perfect circular shape is formed by the pore in the microstructure. For shrinkage pore \( \beta<0.3 \) and for gaseous pores \( \beta>0.3 \). As \( \beta \) decreases, the shapes become more elongated showing a departure from a perfect sphere (Huang and Lu, 2003)

The location \( 1<D<2 \) represent less regular shapes. It was also discovered that the larger the roughness, the more irregular a pore and thus more stress concentration.

Area of the total pore \( A_T = \text{Area of yardstick} \times \text{Number of yardsticks} \)

To calculate the perimeter \( P \) of the pore, the Slit Island Method (SIM) (Bigeralle and Lost, 2006) introduced by (Mandelbort, 1983) is used. It is expressed as

\[ \log_e P = 0.5D \log_e A_T \] (4)

\[ P = e^{0.5D \log_e A_T} \] (5)

Using the Eq. (1), (2), and (5) above, an interactive software in Matlab programming language is developed to obtain the numerical values of the fractal dimension \( D \) and the sphericity \( \beta \) for the microstructures Fig.2, Fig.3, and Fig.4. The flow chart of the Matlab program is Shown in Fig.5.

2.4. Pore Distribution

The pore distribution in the work is studied using the spatial point pattern (SPP). The SPP has been used in ecology, and microbiology due to the inherent clustering nature of certain biological processes. It has also received some attention in material science, with majority of the work concentrating on the study of second phase particles (Vander Voort, 1991; Parse and Wert, 1993; Spitzig et. al., 1985) and characteristics of grains (Kurzydlowski et al., 1994). The SPP can be associated with different types of porosity: (a) regularity- gas or round pores always formed at a distance from their immediate neighbours (b) clustering-shrinkage or irregular pores, always found close to their immediate neighbours with possibility of linking one another (c) clustering on random background- irregular pores on round pore background (Anson and Gruzleski 1999).
Figure 4: Helium implanted specimen pit and pore morphology
Start

S=array of 200 by 200 zeros
A=array of 100 by 100 zeros
B=array of 50 by 50 zeros
C=array of 25 by 25 zeros
Count=array of 1 by 4 zeros

Read image from graphics file into variable rgb

Display the image in rgb

For i=1,2,3…400
  For j=1,2,3…400
    For k=1,2,3
      If the value of rgb(i,j,k)>50
        Change colour of image to black
        Display the image
      Else
        Change colour of image to white
        Display the image
      End If
    End For
  End For
End For

For i=2,4,6…16
  For l=1,2,3…400
    For m=1,2,3…400
      For n=1,2,3
        If the value of rgb(l,m,n) is equal to 255
          Stop
        End If
      End For
    End For
  End For
End For

Stop
If $i=2$

If $i=4$

If $i=8$

If $i=16$

Stop

Yes

No

Yes

No

Yes

No

Yes

Count for grid size 2 pixels-x-2 pixels, count only once per box

Count for grid size 4 pixels-x-4 pixels, count only once per box

Count for grid size 8 pixels-x-8 pixels, count only once per box

Count for grid size 16 pixels-x-16 pixels, count only once per box

Display count result

$x=[200 \ 100 \ 50 \ 25]$

Compute $X=\log_{10}(x)$ and $Y=\log_{10}(\text{count})$

Display the graph of $x$ against $Y$

Fit line to plot and calculate the fractal dimension
Display figure with 200-x-200 grid

X₁=0,2,4,...400
Y₁=0,2,4,...400

Compute Y=X multiply by Array 1 by 201 of ones. Hold the current graph in the figure and plot the graph of X₁ against Y. Also compute X=Y multiply by Array 1 by 201 of ones. Hold the current graph in the figure and plot the graph of X against Y₁

Display figure with 100-x-100 grid

X₁=0,4,8,...400
Y₁=0,4,8,...400

Compute Y=X multiply by Array 1 by 101 of ones. Hold the current graph in the figure and plot the graph of X₁ against Y. Also compute X=Y multiply by Array 1 by 101 of ones. Hold the current graph in the figure and plot the graph of X against Y₁

Display figure with 50-x-50 grid

X₁=0,8,16,...400
Y₁=0,8,16,...400
3. RESULT AND DISCUSSION

The microstructure Fig.2 shows the pores and pits in the virgin specimen of AISI 316LSS with the pores being of irregular shapes (i.e. $\beta<0.3$ and $D$ approaching 2). Table 2 summarizes the values of the fractal dimensions and the sphericity of the selected pores from the virgin specimen of AISI316LSS. The worst of the pores is the pore with $\beta=0.0011$ and $D=1.3584$ while the best of the pores is the pore with $\beta=0.0398$ and $D=1.1136$. In addition, from the porosity distribution map Fig.6, it can be observed that the pores in the virgin AISI316LSS are clustered, thereby making it possible for the pores to link each other and therefore aiding crack propagation.

<table>
<thead>
<tr>
<th>Pore</th>
<th>Sphericity $\beta$</th>
<th>Fractal Dimension $D$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0031</td>
<td>1.4136</td>
</tr>
<tr>
<td>2</td>
<td>0.0108</td>
<td>1.2423</td>
</tr>
<tr>
<td>3</td>
<td>0.0012</td>
<td>1.3869</td>
</tr>
<tr>
<td>4</td>
<td>0.0011</td>
<td>1.3584</td>
</tr>
<tr>
<td>5</td>
<td>0.0089</td>
<td>1.2422</td>
</tr>
<tr>
<td>6</td>
<td>0.0398</td>
<td>1.1136</td>
</tr>
<tr>
<td>7</td>
<td>0.0066</td>
<td>1.2105</td>
</tr>
<tr>
<td>8</td>
<td>0.0044</td>
<td>1.2723</td>
</tr>
</tbody>
</table>
It can be observed that there is a great reduction in the number of pores and pits found in the microstructure of the oxygen-implanted specimen if compared with what is obtained in the microstructure of the virgin specimen. Table 3 summarizes the values of the fractal dimensions and the sphericity of the selected pores from the virgin specimen of AISI316LSS. The worst of the pores is the pore with $\beta=0.0035$ and $D=1.2506$ while the best of the pores is the pore with $\beta=0.4955$ and $D=1.0186$ (i.e. with regular shape $\beta<0.3$). In addition, from the porosity distribution map Fig. 7, it can be observed that the pores in the oxygen implanted AISI316LSS are randomly distributed, thereby making it difficult for the pores to link each other.

<table>
<thead>
<tr>
<th>Pore</th>
<th>Sphericity $\beta$</th>
<th>Fractal Dimension $D$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0035</td>
<td>1.2506</td>
</tr>
<tr>
<td>2</td>
<td>0.4955</td>
<td>1.0186</td>
</tr>
<tr>
<td>3</td>
<td>0.2621</td>
<td>1.0925</td>
</tr>
<tr>
<td>4</td>
<td>0.0433</td>
<td>1.0303</td>
</tr>
</tbody>
</table>
Similarly, it can also be observed that there is a great reduction in the number of pores and pits found in the microstructure of the helium-implanted specimen if compared with what is obtained in the microstructure of the virgin specimen. Table 4 summarizes the values of the fractal dimensions and the sphericity of the selected pores from the virgin specimen of AISI316LSS. The worst of the pores is the pore with $\beta=0.0263$ and $D=1.1571$ while the best of the pores is the pore with $\beta=0.4402$ and $D=1.0667$ (i.e with regular shape $\beta<0.3$). In addition, from the porosity distribution map Fig.8, it can be observed that the pores in the helium implanted AISI316LSS are randomly distributed, thereby making it difficult for the pores to link each other.
The shape, type, and distribution of the pores have a lot to do on the physical properties of AISI316LSS particularly the hardness property considered in this work. The low hardness value of the virgin AISI 316LSS can be attributed to the clustering of the pores in the microstructure of the specimen, while the high value observed for the helium implanted specimen is due to the wide spacing of the pores.

4. CONCLUSIONS
The following conclusion emerged from the analysis
a) The surface hardness is found to be 1202HV for helium implanted and 1020HV for oxygen implanted, while it is found to be 195HV for the virgin material. The hardness of the helium and oxygen implanted samples is found to be increased by about 600% and 500% respectively, when compared to the virgin samples.
b) Ion implantation has changed the shape, type, and distribution of the pores in the virgin AISI316LSS.
c) The pores considered in the virgin specimen are all shrinkage or irregular pores. The “best” of the pores in oxygen and helium implanted specimens are the regular or round pores.

5. REFERENCES