Fatigue properties of a biomedical 316L steel processed by surface mechanical attrition

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Abstract. This work deals with the influence of surface mechanical attrition treatment (SMAT) on fatigue properties of a medical grade 316L stainless steel. Metallurgical parameters governed by SMAT such as micro-hardness and nanocrystalline layer are characterized using different techniques. Low cycle fatigue tests are performed to investigate the fatigue properties of untreated and SMAT-processed samples. The results show that the stress amplitude of SMAT-processed samples with two different treatment intensities is significantly enhanced compared to untreated samples, while the fatigue strength represented by the number of cycles to failure is not improved in the investigated strain range. The enhancement in the stress amplitude of treated samples can be attributed to the influence of the SMAT affected layer.

1. Introduction
Austenitic stainless steel AISI 316 L is used in a wide variety of applications, ranging from nuclear, chemical, petrochemical industries, to marine and offshore, due to its outstanding resistance to corrosion. The medical grade of this alloy is also extensively used in the manufacture of biomedical devices such as implants and equipment (prostheses, orthopedic implants, etc.) due to its excellent bio-compatibility [1, 2], good ductility and toughness. Fatigue properties of 316L stainless steel are of current interest due to its increasing industrial applications.

It is well known that fatigue strength of materials is sensitive to both grain size and residual stresses. On the one hand, nanometer size grains could effectively increase yield strength and enhance fatigue crack initiation resistance. On the other hand, coarse grains exhibit better crack propagation resistance due to good ductility and toughness. Since fatigue cracks generally initiate at a surface and propagate through the material, a component with a nanocrystalline surface (NS) layer and an inner coarse grain matrix can be expected to have improved fatigue strength.

Surface mechanical attrition treatment (SMAT) is a technology which can modify the coarse grained surface layer of a material into nanosized grains by severe plastic deformation (SPD) [3, 4]. SMAT is based on mechanical impacts at high strain rates to introduce a large number of defects such as dislocations and/or deformation twins in order to obtain grains with a nanometer size at the surface. Significant compressive residual stresses can also be present in the nanocrystallized surface generated by SMAT. These high compressive residual stresses coupled with a grain refinement of the microstructure are expected to delay fatigue crack initiation and propagation of the SMAT-processed material and thus to enhance fatigue strength [5].

Some investigations were performed in order to study the fatigue strength of SMAT-processed materials under high cycle fatigue (HCF) such as 316L [5, 6], Ti6Al4V [7], C-2000 [8-10], 718 alloy [11]. The improvement of fatigue strength by appropriate SMAT conditions was obtained. However, a prolonged SMAT leads to an “over-processing”, which induces fatigue resistance degradation rather than improvement [8]. This degradation is due to the presence of micro-damage or crack induced by the shot impacting during SMAT when the treatment intensity is too high [7].
To the authors’ knowledge, the effect of SMAT on fatigue strength under low cycle fatigue (LCF) has not yet been extensively investigated. More particularly, the effect of SMAT on fatigue behavior of materials, such as hardening/softening, is an interesting issue. This kind of study can be crucial while considering the use of SMAT-processed materials in practical applications. The study presented in this paper was undertaken to investigate the fatigue properties of SMAT-processed 316 L stainless steel under LCF and to understand the influence of SMAT on fatigue strength. The analysis of damage mechanisms was carried out thanks to scanning electron microscopy (SEM) observations of fracture surfaces.

2. Material and experimental procedures

The investigated material is an austenitic stainless steel AISI 316 - ASTM F138, whose chemical composition is shown in Tab. 1. The as-received material has an initial grain size of about 10 µm. For the fatigue tests, smooth cylindrical specimens with a reduced section of 6 mm diameter were prepared. SMAT was performed on a part of the fatigue specimens. SMAT is based on the vibration of spherical shot (3 mm diameter, in this work) boosted by a high frequency (20 kHz) ultrasonic generator. Random shot impacts are generated at specimen surface leading to the formation of multidirectional plastic deformation as well as a superficial nanocrystalline layer. In this paper, two SMAT conditions, respectively named High and Very High are considered. SMAT High corresponds to a treatment of 30 minutes with a generator power of 30%. Whereas for SMAT Very High, a treatment of 15 minutes was first performed with a generator power of 30%, followed by a treatment of 5 minutes with a generator power of 50%.

LCF tests were carried out on the untreated and SMAT-processed samples at room temperature using a servohydraulic machine. To investigate cyclic hardening/softening phenomenon, fully reversed tension-compression fatigue tests were performed under strain control (R = −1). An extensometer with a gauge length of 10 mm was employed to control the total strain and a strain rate of $4 \times 10^{-3} \text{s}^{-1}$ was used for all the fatigue tests.

Table 1. Chemical composition (wt.%) of the studied biomedical grade 316 L stainless steel.

|     | Fe   | C    | Mn   | Si   | P    | S    | Cr   | Ni   | Mo   | Cu   | N    | Ti   | V    |
|-----|------|------|------|------|------|------|------|------|------|------|------|------|------|
|     | 48.4 | 0.013| 1.7  | 0.26 | 0.017| 0.003| 17.37| 14.52| 2.80 | 0.08 | 0.088| <0.005| 0.07 |

3. Results and discussion

3.1. Metallurgical state after SMAT

Cross-sectional microscopic observations of samples subjected to SMAT indicate severe plastic deformation at the surface and near surface layers. Fig. 1 shows a SEM observation demonstrating a grain size gradient in microstructure (i.e. finer grains at the surface and near surface regions and coarse grains in the bulk). Plastic deformation of the surface layer by the impingement of flying shot at a high strain rate enabled a progressive refinement of coarse grains down to nanometer scale.

Surface hardness measurements were performed on cross-sections using a Vickers micro-hardness tester to investigate the hardness variation induced by SMAT. Typical hardness evolution curves are shown in Fig. 2. There is a significant hardness increase at the surface of the specimens subjected to SMAT. The depth of the hardened layer is about 400 µm in the treated specimens. The initial hardness (as-received state) is about 240 HV$_{0.025}$ and the surface hardness increases up to about 400 HV$_{0.025}$ after SMAT. The micro-hardness profiles show that the higher the treatment intensity is, the higher the hardness becomes. The hardness increase induced by SMAT is generally due to the increase of grain refinement and strain hardening. In the case of austenitic stainless steels, another possibility is that austenitic phase is partially transformed into martensite under the effect of severe plastic deformation during SMAT [5, 12, 13]. The presence of the martensitic phase induced by
SMAT can lead to a hardness increase of the affected surface layer [13]. It is worth noting that the slight hardness increase at the surface of untreated sample can be due to the machining.

![Cross-sectional SEM micrograph showing a gradient of microstructure for a sample treated by SMAT High [14].](image)

Figure 1.

![Hardness variation with the depth for untreated and SMAT-processed samples under different treatment conditions.](image)

Figure 2.

3.2. LCF hardening/softening behavior

Cyclic hardening and softening behaviors of different sample states are shown in Fig. 3. The cyclic stress response for all the test conditions is characterized by an initial hardening during first several cycles, followed by gradual softening. At lower total strain amplitude, the maximum stress amplitude is reached earlier. For example, the maximum stress amplitude is reached just after one cycle for the total strain amplitude of 0.3%, while it is reached after nine cycles for 0.8%, in the case of untreated samples (Fig. 3a). Long term softening after a short period of initial hardening occurs for untreated samples loaded under low strain amplitudes. When the strain amplitude is higher than 0.5%, secondary cyclic hardening follows the initial hardening and a short period of softening saturation can be clearly observed. This secondary hardening phenomenon is more pronounced as the total strain amplitude increases. At higher strain amplitudes (1.25%, for example), the secondary
hardening is more significant and the stress amplitude increases continuously. It was recently postulated that the secondary cyclic hardening at room temperature might be due to the formation of martensite in 316L stainless steel [15, 16]. It is well documented in the literature that the mechanical behavior of metastable austenitic stainless steels during static and cyclic loading is influenced by the martensite transformation [17-19]. Metastable austenite can undergo progressive strain-induced martensitic transformation during cyclic loading. The extent of martensitic transformation is a function of both the cyclic strain amplitude and the amount of accumulated plastic strain during cyclic loading [15, 20]. The formation of martensite on cyclic straining occurs predominantly at higher strain amplitudes [18].

![Graphs showing stress amplitude vs. number of cycles for different strain amplitudes.](image)
In the case of SMAT-processed samples, the global cyclic hardening/softening behavior is basically the same as the untreated ones. However, for a given total strain amplitude, the stress level reached is significantly higher for SMAT-processed samples. In addition, the secondary cyclic hardening phenomenon becomes less pronounced when SMAT intensity is increased. For example, for the total strain amplitude of 0.8%, there is a slight secondary hardening in the case of SMAT High, while this hardening phenomenon does not occur for SMAT Very High. Moreover, it seems there is a stress amplitude saturation following the cyclic softening (Fig. 4). The samples are strengthened by SMAT since stress levels reached, in particular for SMAT Very High, are significantly higher through the entire fatigue test duration. It seems that SMAT, even if it modifies only the top surface of the sample, could enhance the mechanical strength and change the cyclic hardening/softening behavior of the samples. Moreover, the first cyclic hardening behavior is also modified by SMAT. For untreated samples, the maximum stress amplitude is reached after about ten cycles, while it is reached after only three cycles in the case of SMAT Very High (Fig. 4).

Figure 3. Cyclic hardening/softening curves of 316L for different samples: (a) untreated, (b) after SMAT High, and (c) after SMAT Very High.

Figure 4. Comparison of cyclic hardening/softening behavior for different samples with total strain amplitude of 0.8%.
The difference of stress amplitude between samples subjected to different treatments can also be illustrated by hysteresis loops shown in Fig. 5 which demonstrates the first several cycles until the peak stress amplitude. It can be seen that the stress level reached by SMAT-processed samples is higher than the untreated samples. The stress level increases with the increase of SMAT intensity.

In order to understand how the fatigue behavior of samples was modified, the difference between untreated and SMAT-processed samples should be first clarified. For a sample treated by SMAT, the region near the top surface is subjected to severe plastic deformation. A grain size gradient distribution from a few nanometers (in the top surface layer) to several microns (in the interior) is developed. The core of the specimens is not affected by SMAT, the material characteristics such as grain size remain unchanged as well as its mechanical properties. The different material states for SMAT-processed sample are schematically described in Fig. 6.

Since the core region is not affected by SMAT, its properties should be the same as untreated samples. Thus the difference of fatigue behavior is due to the surface layer affected by SMAT. The stress enhancement can be qualitatively explained as follows. The total depth affected by SMAT is about 0.4 mm and the material properties in this layer vary with the depth, as revealed by the hardness curves (Fig. 2). The diameter of the reduced section of cylindrical
specimens is 6 mm. This means that the sectional surface area affected by SMAT represents about 20% of the total sectional surface area. For SMAT High samples, the average microhardness in the affected layer is about 310 HV_{0.025} (HV_{layer} = 310), while the initial hardness is about 240 HV_{0.025} (HV_{bulk} = 240). It is well known that material strength can be considered as proportional to its hardness. Applying a simple mixing mechanical law, the enhancement in percent can be estimated as:

\[
\frac{[HV_{bulk} \times f_{bulk} + HV_{layer} \times f_{layer} - 1] \times 100}{HV_{bulk}} = 5.8\%
\]

where \( f_{bulk} (=0.8) \) represents the volume fraction of the core of the sample which is not affected by SMAT, and \( f_{layer} (=0.2) \) corresponds to the volume fraction of the SMAT affected layer.

The same calculation can be applied to SMAT Very High for which the average hardness is about 340 HV_{0.025}. The theoretical calculated enhancement is 8.3%, which is consistent with the experimental results corresponding to SMAT Very High condition.

The difference in cyclic hardening/softening behavior can also be related to the contribution of the SMAT affected layer. In this layer, there are a large number of defects such as dislocations and/or deformation twins, and the dislocation density is high. In addition, SMAT induced martensitic phase can be present in this layer. Under cyclic loading, a layer with high dislocation density is expected to significantly undergo cyclic softening since the dislocation annihilation is more rapid than the dislocation generation. Thus in the region where the cyclic softening occurs (in the stress amplitude evolution curves), the samples treated by SMAT manifest a more pronounced cyclic softening behavior (Fig. 4). After this cyclic softening region, a secondary hardening should occur in the core region, which can be due to the martensitic transformation occurring during cyclic loading, as indicated above. However, cyclic softening is expected to continue in the SMAT affected layer, and could create a competition with the secondary hardening which occurs in the core region. This competition may delay the occurrence of secondary hardening of SMAT-processed samples, as demonstrated by the stress amplitude evolution curves shown in Fig. 4.

### 3.3. Fatigue lifetime analysis

Table 2 summarizes the effect of strain amplitude on LCF behavior of different sample states. The values of total strain amplitude \( \Delta \varepsilon / 2 \), and the number of cycles to failure \( N_f \) are included.

| Strain amplitude \( \Delta \varepsilon / 2 \) (%) | Number of cycles to failure \( N_f \) |
|---------------------------------------------|--------------------------------------|
| Untreated | SMAT High | SMAT Very High |
| 0.4    | 12 055   | –          | –          |
| 0.5    | 5 920    | 5 809      | 5 323      |
| 0.8    | 1 122    | 1 365      | 1 172      |
| 1.0    | 607      | –          | –          |
| 1.25   | 423      | 339        | 320        |

The variation of fatigue life with total strain amplitudes is presented in Fig. 7 for untreated and SMAT-processed conditions. It shows that according to the curves of \( \Delta \varepsilon / 2 \) as a function of \( N_f \), there is almost no difference for the three sample states. This means that the two SMAT
conditions have no effect on fatigue strength in the observed range of low cycle fatigue when total strain is controlled. This phenomenon may be explained by the following arguments. Although the stress level is enhanced by SMAT, the total area of hysteresis loops is almost the same for all the three samples (Fig. 5). It is generally considered that the area enclosed in hysteresis loop represents the plastic strain energy density [21]. Thus in the case of these total strain control tests, the energy dissipation rate is expected to be very similar for the three samples. It can be expected that a certain quantity of energy has been introduced by SMAT, and the energy stored in SMAT-processed samples is higher than in untreated samples. However this energy increase induced by SMAT is not very significant since the layer affected by SMAT is thin compared to the diameter of the sample. Consequently, it can be observed that very similar numbers of cycles to failure were obtained for untreated and treated samples. It is important to notice that this explanation is based on the assumption that the cracking mechanism is not changed by SMAT. This point seems to be supported by SEM observations of fracture surfaces. For all the test conditions investigated in this work, crack initiation occurs likely on external surfaces, as shown in Fig. 8.

Finally, compressive residual stress is an important parameter induced by SMAT. It is well known that compressive residual stress may improve the material resistance to fatigue crack initiation and propagation. Nevertheless, previous studies indicated that compressive residual stress only affects fatigue properties under HCF, as residual stress is easily relaxed by high strain amplitude in LCF [22, 23].
4. Conclusion
Based on the results and analyses of LCF tests conducted on untreated and SMAT-processed samples, following conclusions can be drawn:
- The occurrence of secondary cyclic hardening in the case of higher strain amplitude could be attributed to the strain-induced martensitic transformation for the studied 316L steel. Higher cyclic strain amplitude and larger amount of accumulated plastic strain during cyclic loading could promote this martensitic transformation.
- The enhancement in stress amplitude of SMAT-processed samples can be mainly due to the effect of the SMAT affected layer. In this layer, the material strength is increased by introducing a large number of defects and a certain quantity of martensitic phase.
- SMAT has no effect on fatigue strength represented by the number of cycles to failure in the LCF observed range with a total strain control. This is due to the fact that the energy dissipation rate is very similar for the three studied conditions.

The above conclusions remain qualitative. It could be interesting to quantitatively separate the contribution of each constituent such as nanostructured layer, work hardened layer. Future work aims to properly characterize the constitutive behavior of each material layer.

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