Fatigue life improvement through surface nanostructuring of stainless steel by means of surface mechanical attrition treatment

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Abstract

The effect of a nanocrystalline surface layer on the fatigue behavior of a 316L stainless steel is investigated. Significant enhancements of the yield stress and the fatigue limit have been achieved through surface mechanical attrition treatment (SMAT). It is also shown that these mechanical characteristics after SMAT can be significantly improved by the use of a short post-annealing treatment. Such annealing treatment is suggested to cause a recovery at the grain boundaries leading to a reduction of the internal stress.

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1. Introduction

Austenitic 316L stainless steel, due to its excellent corrosion resistance, is successfully used in a wide range of environments such as in the chemical, petrochemical, nuclear and food industries [1]. However, it possesses relatively low strength and poor wear resistance and a very limited number of surface modification techniques can be applied to austenitic stainless steels without causing any loss of their advantageous properties such as corrosion resistance and ductility [2,3]. In recent years, with the emergence of nanocrystalline materials, new ways to improve the mechanical properties of materials have been developed.

With regards to the Hall–Petch relationship, these nanostructured materials have greatly enhanced mechanical properties compared to their counterparts. However, better strength does not necessarily mean better fatigue performance. Results obtained with ultrafine-grained materials, prepared by severe plastic deformation, have shown surprising results, with their fatigue lives being inferior to those of their coarse-grained counterparts [4,5]. Based on experimental work, the mechanism responsible was identified as cyclic softening [5]. The production of bulk nanocrystalline materials has also been revealed as time consuming and expensive.

In fact, most failures of materials occur on their surfaces, such as fatigue fracture, fretting fatigue, wear and corrosion; thus, optimization of the surface microstructure and properties may effectively enhance the global behavior and particularly the service lifetime of materials. An increasing amount of evidence of the novel properties of nanocrystalline materials has been obtained [6,7]. Hence, surface modification by generation of a nanostructured surface layer on a material, referred to as surface nanocrystallization, would be expected to improve the overall properties and performance of materials.

In this paper, the fatigue behavior of a surface nanostructured stainless steel obtained by surface mechanical attrition treatment (SMAT) is investigated. This kind of study can be crucial when considering the use of nanostructured materials in practical applications. So far, the mechanisms for the generation of nanostructures during SMAT...
have been well investigated [8,9] but no study has been carried out on the mechanical and fatigue behaviors of such surface nanostructured materials.

2. Experimental procedures

The material investigated was the austenitic stainless steel 316L whose chemical composition is (in mass%): 0.0019C, 17.07Cr, 11.95Ni, 2.04Mo, 1.68Mn, 0.35Si, 0.04Cu, and 0.07S. The as-received state had an initial grain size between 10 and 50 μm. For the fatigue tests, cylindrical specimens with a reduced section of 6 mm diameter were used.

To achieve surface nanocrystallization on these samples, ultrasonic-assisted SMAT was used, as shown in Fig. 1 [10]. To get a homogenous nanostructured surface a rotating motor was adapted for the SMAT. A treatment time of 15 min and perfectly spherical shot of 2 mm and 3 mm of diameter were employed. Traction compression fatigue tests on the specimens just after SMAT and on those which also had a post-annealing treatment were carried out at room temperature with a standard servohydraulic machine under stress control with zero mean stress (R = −1) and a cycling frequency of 10 Hz.

The microstructure of the specimens with their nanostructured surface layer was observed by transmission electron microscopy (TEM) using a JEOL-2010 electron microscope (operating at 200 kV).

The hardness variation along the depth was measured on cross-sectional samples by using a Nanoindenter (XP™) fitted with a Berkovich diamond indenter. The nanoindenter was calibrated by using a SiO2 standard specimen. The maximum load for the experiments was 20 mN, and the distance between any two neighboring indentations was at least 10 μm.

Residual stress values were measured by X-ray diffraction with a Cu-Kα radiation using the classical sin²ψ method and the (200)-Bragg peak of austenite. To determine the in-depth residual stress distributions, iterative electrolytical removal of thin surface layers and subsequent X-ray measurements were performed. They were also calculated by the incremental hole drilling method [11].

3. Results and discussion

3.1. Microstructure characterization

The microstructure after SMAT on stainless steel was characterized by means of TEM. This study revealed the presence of a 40 μm thick nanostructured surface layer with a grain size of about 20 nm. Fig. 2 shows a TEM observation of the top surface layer after the SMAT of the specimen. This microstructure is characterized by uniformly distributed nanometer-scale grains. At the same time, during the SMA process, a phase transformation from austenite to martensite occurs, leading to a mixed structure as indicated by the corresponding selected area diffraction pattern where martensite phase can be indexed in addition to austenite phase. A previous study on another kind of stainless steel also demonstrated the occurrence of a martensitic phase transformation after SMAT [12].

Analogous to the grain refinement mechanisms during plastic deformation of bulk metals, formation of nanostructures from coarse-grained polycrystals in the surface layer upon the SMAT involves various dislocation activities and development of grain boundaries. In our case, as stainless steel is a material of low stacking fault energy, the plastic deformation mode changes from dislocation slip to mechanical twinning. So TEM observations in the SMAT specimen showed various types of microstructures in the subsurface layer. In the regime of low strain and strain rate, at about 300 μm below the surface, microstructures are characterized by high density of mechanical microtwins with planar dislocations arrays as shown in Fig. 3a. As the strain increases, to accommodate the plastic deformation...

![Fig. 1. Photography of the experimental SMAT set-up.](image1)

![Fig. 2. Bright-field TEM images showing nanocrystal grains in the top surface. Inset is the corresponding SAED indicating the presence of two cubic phases (face-centered cubic and body-centered cubic).](image2)
deformation, twin–twin intersections occur. These intersections of twins are of primary importance in obtaining the nanostructures since they subdivide the original austenite grains into refined blocks. In agreement with results in Ref. [12] and some previous TEM studies, Fig. 3b shows twin–twin intersections observed at roughly 150 μm below the treated surface. Two different sets of twin systems can be observed. The thickness of the twins will be much reduced at extremely high strain and strain rate (close to the surface), feasibly to the nanometer regime. It was also revealed that the martensite phase appears at the intersections of the two sets of mechanical twins, analogous to that observed in the 304 stainless steel during shock deformation [13]. Finally, the formation of randomly oriented nanocrystallites from the refined blocks occurs through processes including grain boundary sliding and/or grain rotation as observed in the case of other processes including severe plastic deformation [14]. So, after the SMAT the size of the grains (or blocks) gradually increases from the nanometer to submicrometer scale as the depth below the surface increases. Since nanocrystalline materials processed with SMAT possess ultrahigh strength [15] and other unusual properties [16], this nanostructured layer may be an important factor in improving the fatigue strength of the material studied.

3.2. Mechanical properties

In earlier work [17], it was observed that the hardness near the surface attains approximately 600–500 Hv, which is a high value that cannot be obtained by conventional surface treatments such as shot peening. This high hardness is mainly due to the presence of the nanostructured layer that follows the Hall–Petch relationship.

Microhardness was measured using nanoindentation and in this case, the hardness reaches about 4.5 GPa at the extreme surface. All the measurements were consistent with the following Hall–Petch relationship [18]: $H_v = H_0 + \frac{K_y}{\sqrt{d}}$ where $H_0$ and $K_y$ are constants equal to 2.32 GPa and 9.5 GPa nm$^{1/2}$, respectively, and this indirectly indicates that the nanostructured layer (grain size less than 100 nm) is about 40 μm thick. Fig. 4 shows the variation of the microhardness along the depth in the SMATed specimen. The high values of microhardness are also due to the presence of a hard phase, the martensite, at the surface of the treated sample as observed from the TEM observations. X-ray diffraction measurements have revealed the presence of a martensitic phase on the surface of the 316L stainless steel. Fig. 5 shows the diffractogram obtained from the nanostructured surface of the stainless steel: body-centered cubic peaks appear very distinctly after SMAT. This phase, which does not exist in the base material, is created during the treatment via the deformations supported by the material. Through successive electropolishing of thin layer by thin layer and followed by diffraction experiments it was seen that the martensite phase is present up to a depth of 200 μm with a decreasing...
quantity as the depth increases. This phase is located close to the region where twin–twin intersections start which corroborates the fact that phase transformation takes place at the crossing of the twins induced by the treatment.

To investigate the effect of the nanostructured layer on the mechanical properties of the stainless steel, tensile tests were done at room temperature. Table 1 summarizes the results obtained with a displacement speed of 2 mm min\(^{-1}\).

For these tests, the samples were treated for different times. The strength increases with the treatment time—this can be explained by the fact that a longer treatment time induces a thicker nanostructured layer. This aspect was confirmed with nanoindentation experiments: when longer times were used, the high microhardness values (4.5 GPa) were maintained along a thicker surface layer. With a treatment time of 30 min, the yield stress becomes equal to 725 MPa, which corresponds to an increase of 141% compared to the yield stress of the base material.

### 3.3. Residual stresses induced by SMAT and their relaxation after annealing

The in-depth residual stress distributions after SMAT and after SMAT followed by annealing at 400 °C for two different times are described in Fig. 6. The incremental hole drilling method as well as the X-ray diffraction (XRD) technique were used to obtain these results. For the incremental hole drilling method, the Cetim-Metro software was used for residual stress evaluation after the calibration coefficients for cylindrical specimen were calculated through development of a 3D simulation. It appeared that by directly using the calibration coefficients available on the Cetim-Metro software (only usable for the planar case) between 5% and 50% error could occur for the result according to the depths. It can be seen that SMA treatment leads to compressive residual stress with important maximum compressive stresses at the surface. The maximum value reaches about −1000 MPa which is very high compared to values obtained with other conventional surface treatments such as shot peening or even deep rolling. As the nanostructured layer formed at the surface exhibits a yield stress of approximately 1500 MPa\(^{[15]}\), such a high residual stress can be reached. Besides, the layer containing compressive residual stress extends to a large depth of about 0.8–0.9 mm from the surface as a consequence of important surface plastic deformations induced by SMAT. The annealing treatment induced residual stresses relax by more than 50% in the near surface regions owing to highest dislocation densities. Basically, this relaxation behavior caused by thermal treatment can be described by the activation energy for self diffusion allowing dislocations to climb and annihilate each other. The higher dislocation density near the surface can explain why the changes are more pronounced at the surface of the specimen. Further increasing annealing time does not lead to a further decrease of the residual stress due to a fast relaxation course of dislocations.

### 3.4. Fatigue strength

The S/N diagram for different treatment conditions is shown in Fig. 7. To prevent the experiments being too time consuming, fatigue tests were continued to a limit of \(N = 2 \times 10^6\) cycles. For fatigue lifetime determination, the criterion of complete separation of the specimen was used except when the lifetime exceeded the limit number of cycles. The fatigue strength of the nanostructured stainless steel is increased considerably compared to the untreated
material and this is not only limited to the high-cycle fatigue (HCF) regime, it can sometimes be the case with ultrafine grain materials prepared by equal channel angular pressing (ECAP) [5]. The fatigue limit of the untreated material is 300 MPa, thus an improvement by 21% is achieved with the nanostructured material prepared with 3 mm diameter shot. In the case where 2 mm diameter shot were used to perform the surface nanocrystallization, the benefit to the fatigue strength is rather low for the high stress amplitude and it becomes more and more accentuated. With the use of 3 mm shot, the increase of the fatigue strength can be observed for both low and high amplitude stress regimes because in such a case, through nanocrystallization treatment, the yield stress is greatly improved and good ductility can still be observed.

In the observed region near the surface two layers can basically be distinguished: a nanocrystalline layer directly at the surface and a layer exhibiting twins and high dislocation densities beneath the surface. Thus, this localized plastically deformed layer leads to formation of high compressive residual stress and refinement of the microstructure (nanostructured layer of high strength) enabling the surface region to exhibit higher resistance against fatigue crack initiation and propagation [19].

By combining the SMA treatment with a post-annealing treatment, the fatigue endurance strength is improved by approximately 5–6% compared with the only nanostructured state. The origin of this phenomenon could be associated with a recovery. Ductility is an important factor that influences the fatigue life of materials in such way that for a material of the same strength, the higher the ductility is, the better the fatigue life will be. In accordance with the mechanical features previously presented, the SMATed stainless steel exhibits an increased strength and a still adequate ductility $\varepsilon_f$, thus an enhanced resistance is observed in the two regimes (HCF and low-cycle fatigue (LCF)). In that way, a suitable annealing treatment leading to an enhanced ductility should even further improve the LCF and HCF resistance.

As observed previously, the ultrafine structure of the surface is characterized by important internal stress and grain boundaries appear to be mostly of high-angle misorientation indicating their highly non-equilibrium character. A high level of defects such as dislocations was also observed in grain interiors and in many regions in the subsurface layer. Therefore, here, our strategy was to relax this internal stress without any grain growth so that a stabilized homogeneous structure could be formed without a loss in strength. In fact, to improve fatigue strength and fracture toughness, one of the main interesting features of processing of nanomaterials is to raise both their strength and ductility. In order to determine the experimental conditions (temperature and time) for recovery, the thermal stability of the nanocrystalline structure was studied. The average grain size was determined by using XRD analyses and TEM observations for the SMATed specimens annealed at various temperatures under vacuum. Fig. 8 shows variations of the average grain size derived from XRD analyses and of the related hardness in the top surface layer (about 5 µm thick) with the annealing temperature. A significant increment in grain size starts at about 600 °C, below which a slight increase of grain size was noticed. This result was verified with TEM observations. When the sample was annealed for 2 h at 500 °C, no grain growth was observed. But then when the sample was maintained at 700 °C for 2 h, an obvious abnormal grain growth occurred resulting in the formation of rather larger grains (>50 nm) with not well-defined boundaries compared to the as-SMAted state. Thus, during annealing, grain growth in the SMATed stainless steel starts at temperatures higher than 600 °C. So parameters for post heat treatment after nanostructuring were chosen below this temperature. At the same time, tensile tests of the annealed samples revealed an important effect of the heat treatment on the overall mechanical behavior of the nanostructured sample. A short annealing between temperature from 300 °C to 500 °C results in a 20% increase in strength combined with enhanced ductility as compared to the as-nanostructured state [17]. So, at
400 °C, the important internal stresses (close to the surface) caused by SMAT are relaxed and an increasing martensite fraction results in higher strength of the subsurface layers inducing a better fatigue behavior. In fact, typically, with annealing treatment, it has been observed that the martensite proportion increases in the near surface region, which represents an additional factor leading to the improvement of the fatigue life of the specimen. Using the 200, 211, 220 reflections for martensite and the 200, 220, 311 reflections for austenite, the volume fraction of martensite was determined to be 15% just after SMAT and to reach a maximum value of 25% after annealing at 400 °C. Similarly to the TEM observations performed on ultrafine grain materials prepared by Valiev’s method (ECAP—high-pressure torsion) [20,21], an ordering of the defect structure at or near grain boundaries may also be suggested through recovery. Thus the improved fatigue resistance in the SMAT specimen where relaxation of compressive residual stresses has occurred appears to be related to the high strength of the nanostructured layer with the transition layer that sustain an important part of the load during cycles. Both the nano-crystalline grain size and high dislocation densities act as obstacles to dislocation slip, so increasing resistance to fatigue crack initiation. Combined with a post-annealing treatment to improve both the strength and ductility of the SMATed sample, it results in an extraordinary increase in both LCF and HCF strength. The improved fatigue life is in this case caused by a combination of strain hardening, strain induced martensitic transformation, a thermally stable nanocrystalline layer and a higher volume fraction of martensite when compared to the as-SMATed state.

4. Conclusions

- A nanostructured surface layer was developed on fatigue specimens of stainless steel with the help of the SMAT. These microstructural changes impede the dislocation movements, delaying crack initiation. The localized plastically deformed surface layer also leads to the formation of high compressive residual stresses enabling a higher resistance against fatigue crack propagation. So a great lifetime improvement is observed as well as an increase in the fatigue strength in the region of LCF and this becomes even more pronounced at HCF.
- Annealing treatment at 400 °C after surface nanocrystallization conducts to recovery and greatly improves the fatigue properties of the material. At this temperature, compressive residual stresses start to relax and the reason for the improved fatigue life is a combination of strain hardening, strain induced martensitic transformation and the nanocrystalline layer that remains stable.

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