FATIGUE LIFE IMPROVEMENT THROUGH NANOSTRUCTURING OF STAINLESS STEEL

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Abstract: In this paper, the effect of a nanocrystalline surface layer on the fatigue behavior of a 316L stainless steel is investigated. A significant enhancement of mechanical properties has been achieved through Surface Mechanical Attrition Treatment (SMAT). The yield stress and the fatigue limit are noticeably increased when compared to those of 316L stainless steel with conventional micrometer grain size. All these results are explained in terms of microstructural investigations, X ray diffraction measurements, tensile and fatigue tests, microhardness measurements and residual stress evaluation through the incremental hole drilling method.

Keywords: SMAT, nanocrystalline surface layer, fatigue lifetime.

1 INTRODUCTION:

Austenitic 316L stainless steel, due to its excellent corrosion resistance, is successfully used in a wide range of environments such as chemical, petrochemical, nuclear and food industries [1]. However, it presents relatively low strength and poor wear resistance, therefore it is very important to improve those properties by applying surface treatment prior to applications. In austenitic stainless steels, the fatigue behavior of materials treated by shot peening [2], laser [3], dynamic ion mixing [4] and coating [5] has been reported but still very limited surface modification techniques can be applied to austenitic stainless steels without any loss of their advantages such as corrosion resistance and ductility. In the recent few years, with the emergence of nanocrystalline materials, new ways to improve the mechanical properties of materials have been developed. In regards to the Hall-Petch relationship these nanostructured materials have greatly enhanced mechanical properties compared to their counterpart component. However, a better strength doesn't necessarily mean better fatigue performances. Results obtained with ultrafine grain materials, prepared by Severe Plastic Deformation (SPD), have shown that the fatigue life was improved for the LCF region but not for the HCF regime [6, 7]. In this paper, the fatigue performances of a nanostructured stainless steel obtained by SMAT are investigated. In particular, this kind of study can be crucial for the use of nanostructured materials in practical applications. So far, the mechanisms of generation of the 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 investigated material is a 316L austenitic stainless steel whose chemical composition is given in table 1. The as-received state has an initial grain size between 10 and 50µm. For the fatigue tests, cylindrical specimens with a reduced section of 6 mm diameter were used. To achieve the surface nanocrystallization on these samples, the process known under the name SMAT was used [10]. To get a homogenous nanostructured surface a rotating motor was adapted to the SMAT. A treatment time of 15 minutes and perfectly spherical shot of 2mm and 3mm of diameter were employed. Traction compression fatigue test on the specimens just after SMA treatment were realized at room temperature with a standard servo hydraulic machine under stress control with zero mean stress (R=-1) and a cycling frequency of 10Hz. The microstructure of the specimens with their nanostructured surface layer was observed by Scanning Electron Microscope after an electrolytic etching under a tension of 2V in a liquid composed of chlorhydric acid (50%) and nitric acid (50%). To demonstrate the presence of nanostructures, TEM observations were realized previously on flat samples submitted to the same treatment conditions as used here [11]. Residual stress values were measured by X ray diffraction with a Cr Kα radiation using the classical $\sin^2\psi$ method and the (200)-Bragg peak of austenite. To determine residual stress depth distributions, iterative electrolytical removal of thin surface layers and subsequent X ray measurements were performed. Stress correction was not carried out. They were also calculated by the incremental hole drilling method.

Element	C	Cr	Ni	Mo	Mn	Si	Cu	S
% wt	0.019	17.07	11.95	2.04	1.68	0.35	0.04	0.07

Table 1. Chemical composition of 316L stainless steel

3 RESULTS AND DISCUSSIONS:

3.1 MICROSTRUCTURE CHARACTERIZATION:

The microstructure after SMA treatment on stainless steel was characterized by means of transmission electron microscope. This study [11] revealed the presence of a nanostructured surface layer with a grain size of about 10 nm on a thickness of about 10 µm from the top surface. Then, the grain size slightly grows along a transition layer of about 90 µm thick until it reaches a classical grain size. The figure 1 shows the cross section of a fatigue specimen obtained after SMA treatment and the figure 2 illustrates the different layers that can be observed in the specimen submitted to the SMA process.

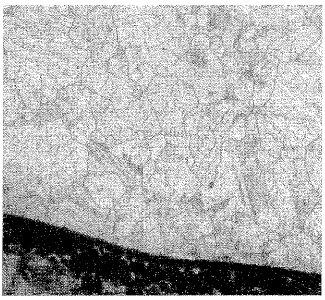

Fig. 1. Microstructure of fatigue specimen after SMAT.

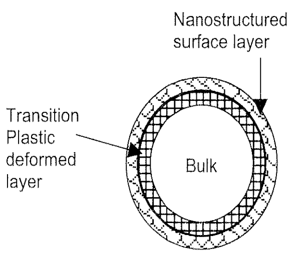

Fig. 2. Schematic representation of the different layers present inside the fatigue specimen (cross section)

Numerous deformation twins are present and seem to indicate the presence of important plastic deformations. With this treatment condition, the three deformation systems, (111) and equivalents, were activated inside several austenitic grains. The multidirectional loadings caused during the treatment are responsible for the activation of these overall sliding systems. Also, these deformation twins are important because they initiate the generation of the superficial nanostructures [11]. Previous work [12] have shown the presence of a nanocrystalline surface layer obtained by shot peening.

3.2 MECHANICAL PROPERTIES:

Vickers hardness profile measured with a load of 0.2kg on the cross section of the SMA treated fatigue specimen is represented in figure 3. It can be seen that the hardness near the surface reaches approximately 500-600 Hv. Such a high hardness can not be obtained by conventional surface treatments such as shot peening that resulted in the maximum value of approximately 300Hv. This high hardness is mainly due to the presence of the nanostructured surface layer that follows the Hall-Pecth relationship. The hardness rapidly decreases when increasing the distance from the surface to finally reach the hardness of the core that is approximately 250Hv.

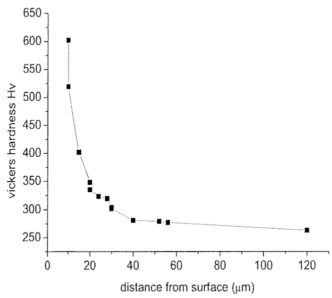

Fig. 3. Hardness depth profile after SMAT

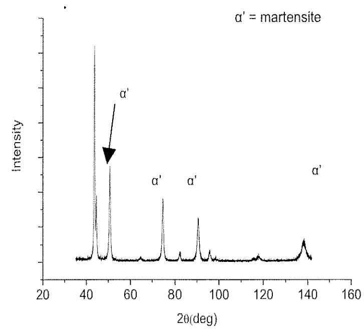

Fig. 4. X ray diffractogram performed on 316L stainless steel after SMAT

Microhardness was measured with the use of nanoindentation and in this case the hardness reaches about 4.5 GPa on a thickness of 40μm from the extreme surface. It was revealed that all the measures were consistent with the following Hall-Petch relationship: $Hv = Ho + Ky / \sqrt{d}$ where Ho and Ky are constants, and this indirectly indicates that the nanostructured layer (grain size less than 100nm) is of 40μm thickness. The high values of microhardness are also due to the presence of a hard phase, the martensite, at the surface of the treated sample. Measures with the help of X-ray diffraction have revealed the presence of a martensitic phase on the surface of the 316L stainless steel. Figure 4 shows the diffractogram obtained on the nanostructured surface of the stainless steel: body-centered cubic peaks appear very distinctly after SMA treatment. This phase, inexistent in the base material, is created during the treatment via the deformations supported by the material.

To investigate the effect of the nanostructured layer on the mechanical properties of the stainless steel, tensile tests were realized at room temperature on tensile sample of 0.5 mm thickness. The table 2 summarizes the results obtained with a displacement speed of 2mm.min^{-1}. For these tests, the samples were treated for different times: 5, 15 and 30 minutes. The strength increases with the treatment time that can be explained by the fact that a longer treatment time induces a thicker nanostructured layer. This aspect was confirmed with nanoindentation experiments:

when a longer SMA treatment time is used, the high microhardness values (4.5 GPa) are maintained along a thicker surface layer.

Treatment time	0.2% yield strength (MPa)	Ultimate tensile strength (MPa)	Total elongation (%)
5 minutes	510	710	43
15 minutes	665	750	22
30 minutes	725	784	15

Table 2. Mechanical properties after SMAT (shot diameter = 3mm)

With a treatment time of 30 minutes, the yield stress becomes equal to 725MPa which corresponds to an increase of 141% compared to the yield stress of the base material.

3.3 RESIDUAL STRESS INDUCED BY SMAT:

The depth distribution of the residual stresses after SMAT is described on figure 5. These results were obtained thanks to the incremental hole drilling method as well as the X ray diffraction technique. For the hole drilling method, the cetim metro software was used for residual stress evaluation after having calculated the calibration coefficients for cylindrical specimen. It appeared that by directly taking into account the calibration coefficients available on the cetim metro software (only usable for plane case) between 5 and 30% error could be made on the result according to the depths. It can be seen that SMA treatment leads to compressive residual stress with important maximum compressive stresses below the surface. The maximum value reaches about -1000MPa which is very high compared to values obtained with other conventional surface treatments as shot peening or even deep rolling. Also, the layer containing compressive residual stress extends to a large depth of about 0.8-0.9mm from the surface as a consequence of important surface plastic deformations induced by the SMA treatment.

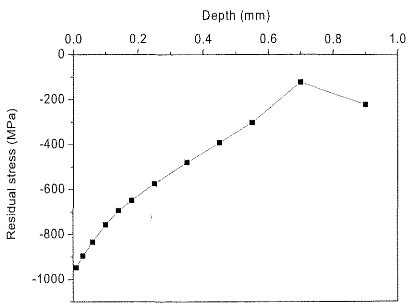

Fig. 5. Residual stress versus distance from surface for fatigue specimen treated with shot of 3mm diameter

3.4 FATIGUE LIFETIME:

The fatigue lifetime improvement for different treatment conditions is shown in figure 6 and figure 7 for an applied stress of 380MPa and of 400MPa respectively. To avoid too much time consuming experiments, fatigue tests were stopped at a limit of $N=2x10^6$ cycles. For fatigue lifetime determination the criteria of complete separation of the specimen was used except when the lifetime exceeded the limit number of cycles. The lifetime of the nanostructured stainless steel is increased considerably compared with the untreated material. In the case where shot of 2 mm of diameter were used to prepare the surface nanocrystallization, the benefit on the lifetime improvement is rather low for the high stress amplitude used here. With the use of 3 mm shot, the effect is more pronounced since through nanocrystallization treatment the yield stress is greatly improved and still good ductility can be observed (table 2).

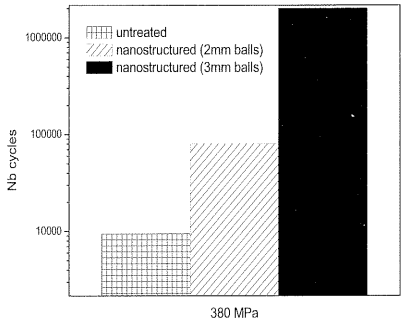

Fig. 6. Lifetime improvement for an applied stress of 380 MPa

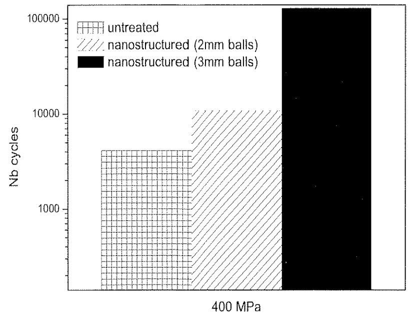

Fig. 7. Lifetime improvement for an applied stress of 400 MPa

3.5 RESIDUAL STRESS RELAXATION THROUGH ANNEALING AND LOADING:

Figure 8 shows the measured residual stress after the treatment of SMA followed by annealing at 400^0C for two different times. On the figure, a spot illustrates the level of the residual stress after a great number of cycles for a nanostructured specimen fatigued with a stress amplitude of 400MPa. This point was measured by XRD. The initial residual stresses due to SMAT have almost disappeared after cyclic loading at stress amplitude of 400 MPa. The apparent residual stress relaxation is caused by cyclic deformation processes with plastic deformation of the core regions of the sample. The annealing treatment induced relaxation of residual stresses by more than 50% in the near surface regions containing high dislocation densities. It also appears that this stress relaxation is rather controlled by annealing temperature than by annealing time, since the residual stress levels after an annealing treatment of 30min or 2h are very close. Basically, this relaxation behavior caused by thermal treatment can be described by the activation energy of self diffusion allowing dislocations to climb and annihilate each other. This can explains that the changes are more visible at the surface of the specimen. These measured residual stress values go in the sense that annealing causes a recovery process and that the nanostructured layer would remain stable. To confirm this, table 3 shows the grain size obtained from XRD measurement on an annealed sample at 400^0C. Thus, an improved fatigue resistance in the SMAT specimen where relaxation of compressive residual stresses has occurred (during cycling) could appear to be related to the high strength of the nanostructured layer delaying crack initiation. With an annealing treatment, we start with a higher equilibrium state which finally could give even better results as it was sometimes observed with ultrafine grain materials.

Phase	Average grains size
Austenitic	12.3 nm +/-3nm
Martensitic	8.1 nm +/-4nm

Table 3. XRD measurement after annealing

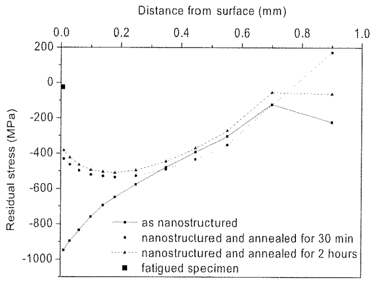

Fig. 8. Stress relaxation after annealing treatment and fatigue test.

4 CONCLUSIONS:

- A nanostructured surface layer was developed on fatigue specimens of stainless steel with help of the SMA treatment. These microstructural changes impede the dislocations movement delaying crack initiation. A great lifetime improvement is observed as well as an increase of the fatigue limit in the region of LCF and it seems becoming even more pronounced at HCF.
- During cyclic loading (tension/compression) complete relaxation of the residual stress induced by the SMAT is observed. An annealing treatment at 400^{0}C seems to lead to a recovery without any grain growth.

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