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Monotonic mechanical properties of plasma nitrided 316L polycrystalline austenitic stainless steel: Mechanical behaviour of the nitrided layer and impact of nitriding residual stresses

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ABSTRACT

The impact of plasma nitriding at 400 °C on the monotonic mechanical behaviour of 316L austenitic stainless steel at room temperature has been investigated. It is shown that the residual stresses in the nitrided layer lead to a tension–compression anisotropy whose magnitude depends on the residual stresses intensity and extension (i.e. thickness of the nitrided layer). Using the stress differential technique, average residual stresses in the nitrided layer ranging from −1.5 up to −3 GPa were measured. The local mechanical behaviour of the nitrided layer has also been investigated through SEM in situ tensile tests. A quasi-brittle mechanical behaviour of the nitrided layer is observed with first evidences of crack initiation for plastic strains below 1%, whatever the nitrided layer. By increasing the total applied strain up to 20%, a progressive segmentation of the layer occurs. The crack initiation location mainly depends on the local nitrided thickness.

1. Introduction

Austenitic stainless steels widely used for their high corrosion resistance present low hardness that limits their use in many applications where severe tribological solicitations can be encountered. As such, surface treatments have been applied to increase surface hardness and to improve the tribological behaviour of these alloys. Plasma nitriding at low temperature is largely used as a surface hardening treatment. Plasma nitriding of polycrystalline austenitic stainless steel [1] performed around 400 °C produces several micrometres thick nitrided layers with increased hardness [2–9], considerably reduced wear rate [1,10–12] and improved fatigue properties [13–15] without any loss of corrosion resistance [7,16–19]. The phase formed within the nitrided layer is usually called the γ_N phase or "expanded" austenite [1]. The introduction of nitrogen into the steel lattice induces a large expansion of the lattice parameter in the subsurface [20,21] and the surface swelling can reach more than 20% [22].

The accommodation of the nitrided layer on its substrate results in residual stresses and consequently to the development of a high plasticity level and subsequent possible damage in the form of cracks [7,23–26]. Moreover a high nitrogen content in the steel substrate can induce a brittle behaviour [26,27] and it is questionable whether the mechanical behaviour of the nitrided layer is chiefly governed by brittleness or by plasticity. Nitrided specimens possess a specific configuration which can be, to some extent, compared to a coating on a substrate, i.e. a nitrided layer on the top of a substrate. In a similar situation, tensile testing of low temperature plasma carburized 316L have been investigated [28]. It was concluded that the cracking behaviour of the treated layer was not as simple as a coating behaviour where the substrate mainly impose its deformation to the coating.

The present work focuses on the characterisation of the mechanical behaviour of the composite-like material, which is nitrided austenitic 316L stainless steel. This is achieved through monotonic and symmetric tension/compression testing investigations, as well as in situ uniaxial tensile test in a Scanning Electron Microscope (SEM) chamber to explore the similarities between the mechanical behaviour of the nitrided and that of a film on a ductile substrate.

2. Material and experimental procedure

2.1. Material

The material investigated was a 316L-type polycrystalline austenitic stainless steel (AFNOR 23 CND 17–12) supplied in the form of a water-quenched rolled plate.

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The specimens for the tension/compression investigation were shaped such as to get a cylindrical gauge with a 3 mm radius and a 10 mm length along the rolling direction RD of the 316L plate. Flat microtensile specimens with a gauge length of 10 mm and a rectangular section (2 mm large, 1 mm thick) were machined by spark erosion for SEM in situ testing experiments. The length direction of these samples also corresponds to RD and their width to the plate transverse direction TD. Samples were then annealed for 1 h at 1055 °C under high vacuum (3×10^{-4} Pa) and finally water-quenched again. Such a procedure confers on the material an average grain size of about 50 μm (70 μm when excluding Σ_3 twins), as determined from large Electron Back-Scattered Diffraction (EBSD) scans. EBSD measurements require the use of well-polished flat specimens with a minimum residual work-hardening. It was obtained from mechanical polishing that combined SiC paper grinding up to grade 4000, diamond spray polishing down to 1 μm followed by mechanical–chemical polishing with 0.05 μm colloidal silica suspension.

2.2. EBSD analysis

The analysis of the plastic activity and of the damage development in individual grains requires the knowledge of the orientation of these grains. It was obtained from EBSD measurements using a JEOL 6100 scanning electron microscope equipped with an orientation imaging microscopy system commercialized by TSL. The specimens were systematically and carefully aligned along one of the axes of the microscope stage, so that the three dimensional crystalline orientation of each grain could be related to the macroscopic axes of the specimens. EBSD scans were performed using a 2 μm hexagonal grid, an acceleration voltage of 25 kV and a beam current typically ~0.2 nA. Under these conditions, the probed depth is about 0.1 μm and the orientation is obtained with an uncertainty close to 0.7°. A representative area of the sample (~0.5 mm^2) containing about 50 grains was investigated before nitriding. Typically, the orientation of a 50 μm sized grain was obtained by averaging ~500 local measurements.

2.3. Nitriding treatment

The 316L specimens were nitrided in the home made URANOS plasma system [29,30]. Plasma was created in a quartz tube by a 13.56 MHz electromagnetic excitation with an incident input power of 700 W. Treatments were performed at a pressure of 7.5 Pa using a 60 sccm N_2 and 40 sccm H_2 mixture. Under these conditions, the ion energy is mainly controlled by the wall furnace temperature and specimens are at floating potential so that surface sputtering can be neglected. The nitriding treatments were carried out at 400 °C for 1 h, 8 h or 33 h such as to get nitrided layers 3.4, 9.1 and 19 μm thick, respectively [22].

2.4. Tension/compression and SEM in situ tensile tests

Monotonic tension/compression tests were performed at room temperature on an INSTRON 1362 electromechanical machine, using the uniaxial push–pull mode. Tests were carried out under total strain rate control mode with two constant strain rates of $\dot{\varepsilon}=8\times10^{-4}\,\mathrm{s}^{-1}$ and $\dot{\varepsilon}=5\times10^{-3}\,\mathrm{s}^{-1}$. The deformation and damage processes under mechanical loading at a microscopic scale were investigated by means of an in situ tensile machine in a scanning electron microscope chamber [31,32]. Monotonic tensile tests were performed at a very low displacement rate of 0.1 mm/min. Successive steps were cumulated to ease the detection of newly created plasticity events and to follow the damage behaviour all along the tensile experiment, within the area where the grain orientations were obtained by EBSD prior nitriding. These tensile tests were made of 16–18 strain steps until a final strain of about 20%. During the tensile experiments, the specimen area under investigation was thoroughly scanned to detect the very first evidences of slip and damage activity. As soon as new events were identified, the global deformation was maintained constant and images were recorded using the Back-Scattered Electron (BSE) mode with a 25 kV acceleration voltage. At the end of a test, the total deformation of the specimen was determined using the displacement of specific marks deposited on top of it prior to testing, allowing to get a rough estimate of $\sim2\times10^{-4}\,\mathrm{s}^{-1}$ for its strain rate.

3. Experimental results and discussion

3.1. Stress–strain curves of the composite-like 316L specimens

Fig. 1 illustrates stress–strain curves obtained for macroscopic tensile (Fig. 1a) and compressive tests (Fig. 1b) for the 8 h and 33 h nitrided specimens. In this figure, the stress and the strain are given in absolute values. As a first observation, the higher the strain rate is, the higher the monotonic properties. This is direct evidence that under such testing conditions, 316L austenitic stainless steel exhibit a viscoplastic behaviour [33], whether a plasma nitriding has been performed or not before testing.

The values of the Young's moduli are reported in Table 1 for the compressive and tensile tests while the yield stresses (defined at

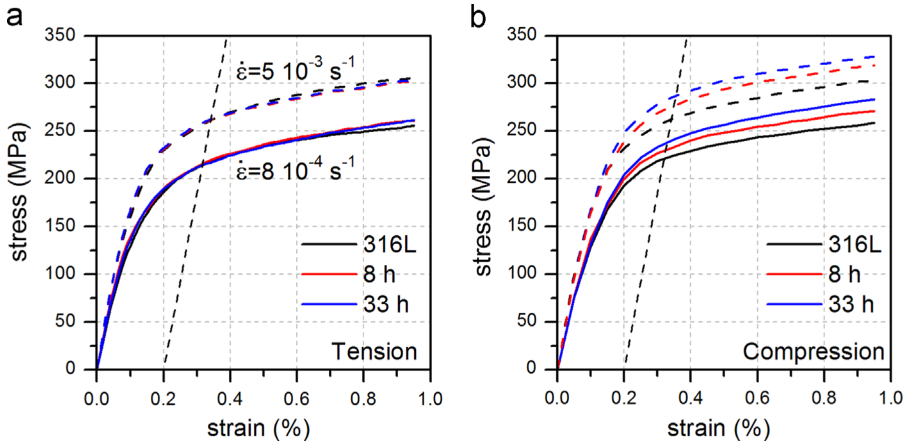

Fig. 1. Tensile (a) and compression (b) monotonic behaviour for untreated, 8 h (8 h) nitrided and 33 h (33 h) nitrided specimens under two strain rates ($8\times10^{-4}\,\mathrm{s}^{-1}$ and $5\times10^{-3}\,\mathrm{s}^{-1}$).