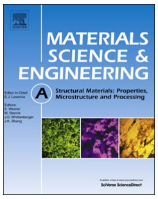
Materials Science & Engineering A 690 (2017) 44–51

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea



Local behavior of an AISI 304 stainless steel submitted to in situ biaxial loading in SEM

C. Caër[*], R. Pesci

Arts et Métiers ParisTech, Laboratoire d'Étude des Microstructures et de Mécanique des Matériaux (LEM3), UMR CNRS 7239, 57070 Metz, France

ARTICLE INFO

Keywords:
Biaxial tensile test
In situ mechanical testing
Electron back scattered diffraction
304 stainless steel
Coarse grains
Martensite

ABSTRACT

The microstructural response of a coarse grained AISI 304 stainless steel submitted to biaxial tensile loading was investigated using SEM and X-ray diffraction. The specimen geometry was designed to allow for biaxial stress state and incipient crack in the center of the active part under biaxial tensile loading. This complex loading was performed step by step by a micromachine fitting into a SEM chamber. At each loading step FSD pictures and EBSD measurements were carried out to study the microstructural evolution of the alloy, namely grain rotations and misorientations, stress-induced martensite formation and crack propagation. According to their initial orientation, grains are found to behave differently under loading. Approximately 60% of grains are shown to reorient to the [110] Z orientation under biaxial tensile loading, whereas the 40% left undergo high plastic deformation. EBSD and XRD measurements respectively performed under loading and on the post mortem specimen highlighted the formation of about 4% of martensite.

1. Introduction

The drive toward miniaturized systems in various engineering fields motivates the study of materials behavior close to their industrial scales. Moreover most of the phenomena occurring at the macroscopic scale result from mechanisms (phase transformation, cracking,..) happening at the microscopic scale.

Numerous experimental means and techniques have therefore been adapted from macroscopic to nanometric scale. This allows to study the influence of environmental constraints such as mechanical loading [1,2], temperature [3,4], speed [5,6] or even irradiation [7,8] on the material behavior at a small scale [9]. Numerous studies can be found in the literature regarding the microstructural evolution of materials under in situ uniaxial tensile tests [10,11]. However, during their real service life, materials are usually loaded according to far more complex loading paths than uniaxial tension. Different mechanical loading paths available on macroscopic tests benches (bulge test [12], bending [13], …) were therefore adapted to smaller scales.

The present work aims at studying microstructural features evolution under a specific complex mechanical loading, namely equi-biaxial tensile loading, using laboratory facilities. This kind of study has already been developed for Synchrotron [14,15] or neutron diffraction [16,17]; however it is important to set up such a loading path in more ordinary equipment such as Scanning Electron Microscopes (SEM). This work involves an approach that combines Electron Backscattered Diffraction (EBSD) and X-ray diffraction (XRD).

The second section of this paper focuses on the presentation of experimental details, advanced techniques and materials used in this work. AISI 304 steel was used in this study as it is the most extensively used steel finding applications in the automotive and nuclear industries in particular. In situ experiments were performed using a miniature biaxial tensile test bench fitted in the SEM and under X-ray diffraction.

Although reference standards for uniaxial tension [18] or bending [19] impose to use specific specimen geometries, the current standard for biaxial tensile tests [20] is less constraining by allowing the use of alternative specimen geometries [21,22]. The third part of this paper therefore presents a convenient specimen geometry determined from Finite Element Method (FEM) simulations to in situ validation in SEM. The newly designed sample geometry should allow for a biaxial stress state in the active part of the specimen and crack initiation in the center of said active part.

From these in situ biaxial tensile tests performed in SEM, the study of mechanical and microstructural features evolution under a complex loading path was performed. Microstructural evolution was first observed according to features such as grain reorientations and misorientations under loading. A particular focus was also made on the stress-induced martensitic transformation. Finally this study focuses on crack initiation and propagation.

---

[*] Correspondence to: Arts et Métiers ParisTech, 4 rue Augustin Fresnel, 57070 Metz, France.
E-mail address: celia.caer@gmail.com (C. Caër).

http://dx.doi.org/10.1016/j.msea.2017.02.087
Received 2 November 2016; Received in revised form 13 February 2017; Accepted 23 February 2017

Table 1
Chemical composition of the AISI 304 stainless steel (% weight).

C	Mn	Cr	Ni	Mo	Cu	Si	Nb
0.006	1.54	18.47	8.3	0.3	0.37	0.48	0.027

2. Materials and methods

2.1. Material processing and preparation

The investigated alloy is an austenitic AISI 304 stainless steel rolled in cylinders of 100 mm diameter; the chemical composition is given in Table 1. This material showed in the as-received state a yield strength σ_y=325 MPa, a maximum strength σ_m=1000 MPa and a Young modulus E = 200GPa [23]. The as-received material was examined using EBSD which showed a too fine-grained microstructure for the study to be carried out. The steel cylinder was therefore annealed in vacuum at 1200 °C for 1 h to increase the grain size up to an average of 300 µm.

Thanks to electro-discharge machining, slices were cut out of the steel cylinders and samples were machined from the slices.

Prior to any EBSD analysis, sample surfaces were carefully prepared. Specimens were first mechanically polished with decreasing SiC papers and diamond particle pastes. The final mechanical polishing was performed with a 1 µm diamond paste. The samples were finally electropolished in a solution of ($C_2H_4O_4$ + H_2O) at 16 V for 1 min.

2.2. Biaxial tensile testing

Samples for macroscopic biaxial tensile tests were mounted on a servo-hydraulic multiaxial testing machine developed by INSTRON. The loading was chosen to follow a balanced biaxial tensile loading path ($\varepsilon_{TD}/\varepsilon_{RD}$=1) path up to failure. The experiments were conducted using a constant strain rate of $1.5*10^{-2}\ s^{-1}$. An infrared camera was placed in front of the specimen in the biaxial tensile bench in order to follow the temperature evolution and the associated stress concentrations during the whole loading.

In situ mechanical tests were performed thanks to a micromachine (model called "Proxima") developed by the company MICROMECHA. This machine, dedicated to mechanical testing in SEM and with XRD (Fig. 1), was fitted with a tensile biaxial set up for this study. In situ equi-biaxial tensile tests have been performed at low strain rates around $1.5*10^{-3}\ s^{-1}$. This experimental setup allows for:

- The quantification of the amount of martensite formed during loading when placing the post-mortem specimen on a XRD goniometer.
- The study of the microstructure evolution (texture, intragranular misorientations, plasticity, phase transformation…) during loading when mounting the micromachine into a SEM.

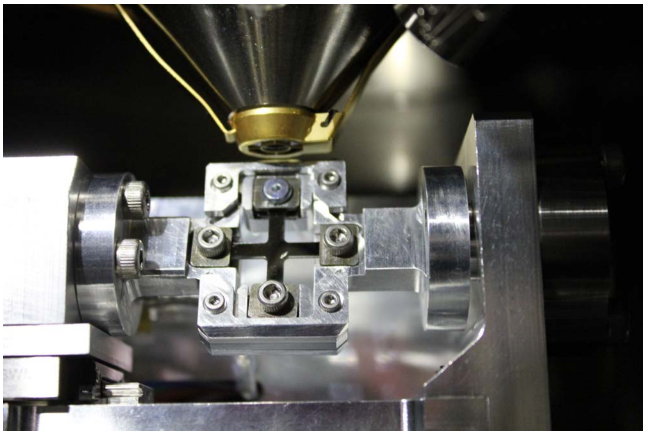


Fig. 1. Micromachine from MICROMECHA company (Proxima model) mounted in a SEM and fitted with the tensile biaxial setting.

Table 2
Planes analyzed and corresponding 2θ angles for both austenite and martensite.

Phase	Austenite		Martensite	
Plane analyzed	{200}	{220}	{200}	{211}
2θ angle (deg)	79	128.7	107.1	154.1

2.3. X-rays diffraction technique

XRD is a very efficient non-destructive technique which offers the opportunity to not only study crystallographic texture but also accurately quantify the volume fraction of phases and evaluate the level of residual stresses. XRD has been used in this study to measure the volume fraction of both austenite and martensite phases in the post-mortem specimens following the ASTM E975-13 standard.

The volume fraction quantification of the two phases contained in AISI 304 steel has been performed with a PROTO iXRD goniometer fitted with a 1D detector by taking into account the intensity of the diffraction peaks of both austenite (A) and martensite (M); two peaks of each phase were retained as summed up in Table 2. Because of the two-phase microstructure of the steel, the sum of the volume fractions of austenite V_A and martensite V_M should be equal to 1.

The volume fraction of austenite is obtained according to (Eq. (1)):

$$V_A = \left(\frac{1}{q}\sum_{j=1}^{q} I^{Aj}/R^{Aj}\right) / \left(\frac{1}{p}\sum_{i=1}^{qp} I^{Mi}/R^{Mi}\right) + \left(\frac{1}{q}\sum_{j=1}^{q} I^{Aj}/R^{Aj}\right) \qquad (1)$$

where I^{Mi} and I^{Ai} are respectively the diffraction peak intensities of martensite and austenite. R^{Mi} and R^{Ai} are coefficients depending on the Lorentz Polarization and the Debye-Waller factors, multiplicity of the considered {*hkl*} reflections and the crystal lattice of the phase [24].

The volume fraction of both austenite and martensite have been measured using a collimator 1 mm in diameter and a chromium tube (λ=2.29 Å) which allows a penetration depth of about 10 µm. The uncertainty in the phase fraction measurement is about 0.3%.

2.4. Scanning Electron Microscopy

The device used in this study is a 7001 Field Electron Gun Scanning Electron Microscope from JEOL equipped with an Oxford EBSD CCD camera fitted with FSD detectors. EBSD data were post-treated with the software CHANNEL 5 from Oxford [25].

Given the coarse grain size, step size was set to 1 µm and the voltage to 15 kV. The eventual field of view obtained was 1.2*1.2 mm.

The biaxial tensile micromachine was set in the SEM and the loading was applied step by step until specimen failure. 15 steps were applied up to failure. SEM images, ESBD mappings and FSD images were performed at each loading step allowing for the microstructure characterization at each stop-off. An effort was made to keep the same area, namely the center of the specimen, under the field emission gun during the whole loading.

The EBSD technique was operated to study the evolution of [26]:

- grain reorientations and misorientations,
- phase transformation as the software was required to identify the crystal lattice of austenite (FCC) and martensite (BCC).

3. Specimen geometry design for in situ loading in SEM

This section details the process of specimen geometry design for biaxial tensile experiments. This specimen geometry should allow for: