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Evaluation of the strain-induced martensitic transformation by acoustic emission monitoring in 304L austenitic stainless steel: Identification of the AE signature of the martensitic transformation and power-law statistics

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ABSTRACT

The aim of the present investigation is to characterize plasticity-induced martensite formation of metastable austenitic stainless steel at room temperature. Acoustic Emission (AE) monitoring is performed on a 304L austenitic stainless steel during fatigue tests. This work aims at identifying the acoustic emission signals associated with the formation of the strain-induced martensite. The present work includes the study of the influence of the specimen geometry. The use of statistical pattern recognition allowed the identification of the acoustic emission signatures for three mechanisms: dislocation motion, mechanical damage and martensitic transformation (MT). Moreover statistics on the energies of the AE signals were found to obey power laws ($P(E) \sim E^{-\alpha}$) with exponent $\alpha = 1.75 \pm 0.15$ for the cluster associated with martensite formation.

1. Introduction

The austenitic phase of some stainless steels is metastable at room temperature, such as that of 304L alloy. Martensitic transformation (MT) γ austenite $\rightarrow \alpha'$ bcc to slightly tetragonal martensite can be induced by quenching or by plastic deformation. By quenching the process starts at temperature Ms. Transformation can also occurs above Ms under the influence of mechanical stresses and strains [1–12]. Cyclic strains also induce some MT. Since the displacive MT modifies the volume (with $\gamma \rightarrow \alpha'$ martensite a volume expansion occurs [2]), internal stresses are generated that can affect the lifetime of components subjected to fatigue. Thus the monitoring of MT is of societal interest and many non-destructive techniques have been applied, among which eddy current testing [13,14] and acoustic emission [15,16]. The aim of this study is to investigate MT by AE and MT in 304L steel during fatigue tests, using two kinds of analysis: pattern recognition and power-laws statistics, which we shortly describe hereunder.

2. Acoustic emission and martensitic transformation

It is well known for a long time that martensite formation in steels can be detected by AE monitoring [17,18]. Since then considerable work has been carried out to relate the detected AE signals to the kinetics of the martensitic transformation [19,20]. The AE of the thermally induced MT has been more frequently investigated [21–23] in the literature than that of the strain-induced MT. The acoustic emission (AE) technique is an efficient way to monitor damage growth in both laboratory specimens and structural components. It deals with the analysis of transient elastic waves generated by a sudden release of energy from localised sources within a material. Sensors set on the specimen surface capture these waves. Then, the recorded signals depend jointly on the events they originate from, and on the elastic and damping properties of the propagation medium and the sensor features. Thus, there is no universal signature of AE events. However, in permanent set-up conditions, similarities exist among AE signals originating from similar events. In the case of ASI 304L steel, many mechanisms during fatigue tests have been confirmed as AE sources including dislocation motion, formation of martensite, twin formation, inclusion fracture, crack nucleation and propagation. Then, discriminating the types of source mechanisms responsible for the detected AE signals is an exciting challenge. Indeed, it could allow to continuously monitor the damage progression in vulnerable

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components. However, two main reasons make the discrimination of the AE mechanisms a non-trivial task. On one hand, the AE signals are complex objects that must be characterized by multiple pertinent descriptors in order to be processed. On the other hand, the acoustic signatures of AE events are not known *a priori* and they are rather scattered due to their sensitivity to the above-mentioned conditions. Consequently, AE signals collected from tests performed in actual conditions must be segmented into clusters based on closest similarities [24–26]. Any AE signal can be represented in an n-dimensional space by a vector which components are the signal descriptors. Cluster analysis partitions the input data space into k regions based on the distances between objects. The number k of clusters may or may not be known *a priori*. Depending on whether the cluster features are known or not, the classification algorithms are said supervised or unsupervised, respectively. In the case of AE data set, features of signal clusters are not available and unsupervised classifiers have to be used in order to search for them in the structure of the data set itself. The k-means algorithm [26,27] is one of the most widely used methods to determine cluster solutions for a particular user-defined number of clusters.

Power law distributions of event sizes are usually observed in physical systems close to the critical point of a second order phase transition. However, power-law behaviour can also occur in driven physical systems with intrinsic disorder, even when the physical process involves a first-order transition. Experimental observations of this kind have been reported for a large variety of phenomena such as earthquakes, martensitic transformation, Barkhausen noise, or rupture [28–31]. In some cases, it has been suggested that such systems follow the theory of Self-Organized Criticality (SOC) and thus evolve until a critical state is reached. Then events occur in avalanches showing no characteristic temporal and spatial scales. An important feature of such systems is that they respond to external perturbations by avalanche of all sizes S with a power law distribution $P(S)\sim S^{-\tau}$ where τ is a scaling exponent. A martensitic transformation is a diffusionless first-order phase transition. It involves a cooperative and almost simultaneous movement of atoms from parent to product phase. In athermal martensite, the martensite phase nucleates from isolated regions which are usually defects like dislocations or grains boundaries. Thus quenched disorder plays an important role in the initial kinetics of athermal martensite. The avalanche size distributions have been mostly investigated through the amplitude and duration of the peaks in acoustic emission signals [31,32]. The important result is that both quantities exhibit power-law distributions. This is an indication that the system evolves without characteristic time and length scales which is a typical feature of criticality. Vives et al. [31] have characterized the avalanche distributions during a thermally induced thermoelastic martensitic phase transition of a Cu–Zn–Al alloy, using AE techniques and shown it exhibits power-law behaviour for more than one decade. They obtained the exponents for the distributions of amplitudes $P(A)\sim A^{-\gamma}$, $\gamma = 3.6 \pm 0.8$ and durations $P(D)\sim D^{-\tau}$, $\tau = 3.5 \pm 0.8$. On the other hand, for stress-induced transformation, Carrillo et al. obtained $\gamma = 2.3 \pm 0.2$ [32]. In the study of dynamical systems, dissipated energy is usually used as a universal and relevant parameter to characterize the size of an instability. The energy E involved in the process is proportional to the square of amplitude, so these studies would lead to energy distributions $P(E)\sim E^{-\alpha}$ with the following expected values of α ($\alpha = (1+\gamma)/2$): $\alpha = 2.3 \pm 0.4$ for thermal cycling [31] and $\alpha = 1.65 \pm 0.1$ for stress cycling [32]. More recently, a theoretical work [33] has been able to reproduce the magnitude of the exponent for thermal cycling but there is no model that can account for the exponent observed in stress cycling yet.

Table 1
Chemical composition of the 304L steel (wt%)

C	0.028
Si	0.490
Mn	1.40
P	0.034
S	0.017
Cr	18.21
Mo	0.46
Ni	8.15
Co	0.11
N	0.045

3. Material and experiments

The material studied in this work is a commercial 304L austenitic stainless steel, previously monitored by Pasco et al. [34] by Barkhausen noise (same batch of material). The material alloy is supplied in the form of a bar, 20 mm in diameter. The chemical composition of the material is given in Table 1. In order to obtain a homogeneous microstructure the material is annealed above 1000 °C during about 1 h and quenched in water. After this treatment, X-ray diffraction enabled to detect 15% of a bcc phase which can be a mixture of ferrite and martensite and 85% of the austenite (for low carbon contents, martensite has bcc structure). The resulting mean grain size is about 50 μm. An estimate of the temperature at which plenty of strain-induced martensite forms is the M_{d30} temperature, corresponding to the temperature at which an actual 30% strain induces the formation of 50% of martensite α'. The Pickering relation (1), given in mass percentage, enables to estimate M_{d30} from the steel composition [34–36]:

$$M_{d30} = 497–13.7Cr\%–20Ni\%–8.1Mn\%–9.2Si\% \\ –462(C + N)\%–18.5Mo\% \quad (1)$$

For the studied steel, the application of the Pickering relation gives a M_{d30} equal to 26 °C which indicates that a martensitic transformation can appear under cyclic loading at room temperature. The dimensions of the fatigue specimen are shown in Fig. 1. Specimens with a cylindrical gauge length of 20 mm and a diameter of 4 mm are machined from the bars. Moreover specimens with a rectangular gauge length of 20 mm are tested in order to allow eddy current measurements [37,38].

Low cycle fatigue tests are performed with alternating load ($R = -1$) at room temperature on a 10 kN hydraulic testing machine. The fatigue tests are carried out under stress control at imposed maximal stress σ_{max} equal to 500 MPa, 600 MPa and 700 MPa with a frequency of 0.25 Hz, 0.166 Hz and 0.0121 Hz. The yield strength of the material is in a range 350–400 MPa. There is a soft transition between elasticity and plasticity for such a material. The strain is continuously measured throughout the test by an extensometer with 8 mm gauge length clamped to the specimen.

Metallography is performed on cuts of fatigue specimens. They are first ground on silicon carbide paper using 80, 180, 400 and 1200 grit. Specimens are further polished with 3 μm diamond paste. The chemical etching is mainly made by dipping the sample in an oxalic acid solution in order to reveal the austenic grain structure including twins, slip bands and martensite. The microstructure of the materials is shown in Fig. 2a. After the fatigue tests the specimens are sectioned parallel and perpendicular to the stress axis. Twins are detected in the specimen before testing.