Materials Science & Engineering A 660 (2016) 225–240

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Insights into dynamic strain aging under cyclic creep with reference to strain burst: Some new observations and mechanisms part-II: Microstructural aspects

Aritra Sarkar [a,*], A. Nagesha [a], P. Parameswaran [b], S. Murugesan [b], R. Sandhya [a], K. Laha [a]

[a] Mechanical Metallurgy Division, Indira Gandhi Centre for Atomic Research, Kalpakkam, 603102 Tamil Nadu, India
[b] Materials Synthesis and Structural Characterization Division, Indira Gandhi Centre for Atomic Research, Kalpakkam 603102, Tamil Nadu, India

ARTICLE INFO

Article history:
Received 27 October 2015
Received in revised form
8 February 2016
Accepted 14 February 2016
Available online 24 February 2016

Keywords:
Strain burst
DSA
Cyclic creep
Planar slip
Twin
316LN SS

ABSTRACT

Cyclic creep behavior of 316LN austenitic stainless steel (SS) was investigated at 823 K at different combinations of mean stress (σ_m), stress amplitude (σ_a) and stress rate. Characteristic strain bursts were observed being attributed to a pronounced influence of dynamic strain aging (DSA). Detailed microstructural investigation carried out through transmission electron microscope (TEM) revealed that dislocation substructure evolving under a process of strain burst during cyclic creep mainly consists of planar deformation bands. The number density of bands was found to be strongly sensitive to σ_m- σ_a -*stress rate* combination employed. An important substructural feature found in this study was the formation of microtwins. Either planar slip or twinning was found to dominate the substructure depending on the loading combination, which was demonstrated through a dislocation distribution map. Dislocation substructure was further correlated with evolution of surface relief studied through atomic force microscopy (AFM) and field emission gun-scanning electron microscopy (FEG-SEM), which depicts the formation of slip markings and nucleation of cracks from persistent slip markings during the course of a strain burst. Finally, well-known theoretical models explaining the mechanism of DSA during tensile deformation were suitably modified for load-controlled scenario and the origin of strain burst as a function of σ_m or stress rate was explained based on the same. Dislocation density measurements were carried out for specimens undergoing strain burst during cyclic creep, which was utilized for reconstituting the models.

1. Introduction

Dynamic strain ageing (DSA) manifests itself in the form of strain bursts under stress-controlled asymmetric cycling referred to as cyclic creep [1]. Several theoretical models have been developed to explain the mechanism of occurrence of serrations appearing during DSA [2–6,13–17]. However, the theories are mostly based on studies carried out through tensile tests. Such investigations also resulted in identifying the strain rate–temperature combinations for the occurrence of DSA pertaining for different alloy systems [1,3,5–10]. It may be noted that manifestations of DSA (onset of serrations) are most prominent at some specific combinations of mobile dislocation velocity and rate of solute diffusion, which in turn are respectively governed by the applied strain rate and temperature [1,3,5–10]. Serrations in the stress-strain hysteresis loops have been shown to appear at a lower temperature range under LCF cycling by virtue of an increased vacancy concentration and consequent acceleration in the rate of diffusion of solute atoms [11,12]. An increase in the cyclic work hardening, reduction in the plastic strain range and increase in the cyclic stress response with a decrease in strain rate or increase in temperature are other manifestations of DSA observed during LCF tests [11,12,18–23]. Investigations pertaining to DSA under cyclic creep of austenitic stainless steel are limited. However, other alloy systems have received some attention. Lorenzo and Laird [23,24] have indicated that strain bursts occurring in polycrystalline copper at room temperature were caused by cyclic latent softening due to cross-slip of screw dislocations during the unloading part of the cycle. Neumann et al. [25] attributed these strain bursts occurring in case of pure metals to the formation of dislocation dipoles during cyclic deformation. However, those investigations are carried out at ambient temperature.

A detailed study has been conducted by Sarkar et al. [26–29] to investigate the manifestations of DSA under cyclic creep in type 316L(N) austenitic stainless steel, the currently favored structural material for the primary side components in sodium-cooled fast

* Corresponding author.
E-mail address: aritra@igcar.gov.in (A. Sarkar).

http://dx.doi.org/10.1016/j.msea.2016.02.044
0921-5093/

reactor (SFR). The nature of strain accumulation under such asymmetric loading is complex, and is a strong function of the stress amplitude (σ_a), mean stress (σ_m) and the rate of applied stress. A thorough investigation on the occurrence of DSA-induced strain bursts under different combinations of σ_m, σ_a and stress rate was carried out by the authors at 823 K which is elaborated in Part-I of this study [1].

The present paper aims to establish the micromechanisms of strain bursts under different loading combinations used in cyclic creep. The evolution of substructure at elevated temperature, especially under the influence of DSA may be totally different. This is explored in the present study through detailed transmission electron microscopy (TEM) investigations. The micromechanism of strain burst is elucidated through an in-depth analysis of the evolution of dislocation substructure by interrupting specimens at various cycle fractions corresponding to strain bursts. Occurrence of strain burst is bound to influence the cyclic slip localization and associated surface slip markings. Hence, a detailed study on the evolution of surface relief and short cracks was carried out through field emission gun-scanning electron microscopy (FEG-SEM) and atomic force microscopy (AFM) on the specimens interrupted at various stages of cyclic creep appertaining to the occurrence of strain bursts. The physical nature of deformation during strain burst was subsequently correlated with the evolution of dislocation patterns and surface relief, with a view to gaining a deeper understanding of the micromechanism associated with strain bursts. Further attempt was initiated towards interpreting the origin of strain bursts in the light of theoretical models on DSA which are mentioned earlier.

2. Experimental

The experimental and specimen details are elaborated in Part-I of this paper [1]. Failed specimens subjected to cyclic creep at 823 K were examined using different characterization techniques viz., FEG-SEM, XRD, AFM and TEM to understand the nature of deformation under such loading. Assessment of surface morphology and short cracks was made using FEG-SEM and AFM (NT-MDT Solver Pro ECAFM) on longitudinally cut sections of the cycled specimens at location around 5 mm away from the fracture surface within an area of 5μ × 5μ. Change in the surface relief due to formation of slip markings during the process of cyclic creep was analyzed using AFM, with the help of NOVA Image Analysis Software (1.0.26.1443). Dislocation density was measured through XRD along the cross-sections of the cycled specimens using a XRG-3000 diffractometer with a curved position sensitive detector using Cu Kα1 radiation of wavelength 1.54056 Å with step size of 0.012° from 20 to 100° (two theta). The dislocation density was evaluated using modified Williamson–Hall equation. The substructural changes occurring during the course of cyclic creep were studied by TEM (CM 200, M/s FEI, The Netherlands) using samples obtained from thin slices, cut at a distance of about 2–5 mm away from the fracture surface. They were first mechanically polished down to about 70 μm followed by electropolishing using a solution containing 20% perchloric acid and 80% methanol at 243 K using a 20 V dc supply.

3. Results and discussion

3.1. Evolution of surface damage under strain burst occurring in cyclic creep

The evolution of surface morphology during the process of strain burst (σ_m: 125 MPa, σ_a: 230 MPa, stress rate: 50 MPa/s) under cyclic creep was studied through AFM and FEG-SEM for specimens interrupted at different fractions of life (10%, 40%, 70% and 100%) which is represented in Fig. 1(b)–(f). Fig. 1(a) indicates the actual cyclic creep curve showing the points of interruption. Surface damage under cyclic loadings typically comprises of slip lines or slip bands. 3-D surface morphology of virgin specimen is represented by the AFM image in Fig. 1b showing minute undulations ranging up to a maximum height of about 100 nm. It may be noted that typical surface finish of specimens used for these experiments varied in the range, 0.1–0.2μ.

The SEM and AFM images of the specimen interrupted after a life fraction of 10% in the above (σ_m: 125 MPa, σ_a: 230 MPa, stress rate: 50 MPa/s) loading condition are presented in Fig. 1c (i) and (ii) respectively. The SEM image shows that surface relief comprises mostly of fine slip markings. Cyclic slip localization led to the development of a few deeper slip markings some of which appear to have transformed into minute cracks (marked by arrows). Even though DSA is active, significant strain gets accumulated in the 1st cycle, as shown in Fig. 1a (referred to as the '*initial strain*' in Part-I) [1]. Besides, progressive cycling causes strain localization which results in a *peak and valley* type morphology on the surface as shown in the AFM image presented in Fig. 1(c) (ii). The number of peaks found under this loading condition greatly surpassed that found in the untested condition (Fig. 1a). Evidence of formation of deeper slip markings and nucleation of secondary cracks is also reflected from the corresponding surface profile obtained from AFM (Fig. 1(c)(iii)) which shows the maximum height and width of the peaks to be ~120 nm and ~0.25 μm respectively as compared to 100 μm for untested specimen (marked in the figure).

The surface relief for the specimen interrupted at 40% of life-fraction is presented in Fig. 1(d)(i) (SEM image) and 1(c)(ii) (AFM image). The SEM image showed a significant increase in the number density of slip markings compared to earlier case (Fig. 1c (i)); the slip markings are mostly found to be persistent as reflected by the presence of extrusions (white bulging along the slip markings) [30,31] (marked by white arrows in the figure). Occurrence of a strain burst within 10–40% of cycling (Fig. 1a) resulted in a sudden accumulation of creep strain which gets further localized into slip markings, making them persistent. Increase in the number of persistent slip markings (PSM) accelerates the process of crack nucleation and pre-existing cracks were found to have grown further (marked by black arrows). The same is corroborated through the AFM image (Fig. 1d (ii)) showing similar *peak and valley* surface morphology as found in Fig. 1(c) with occasional larger peaks. A deep valley is found running across the surface (marked in Fig. 1d (ii)) which is indicative of crack. The corresponding surface profile presented in Fig. 1d(iii) depicts that maximum peak height increased up to ~200 nm and the peak width is ~0.5 μm. This marks an almost two-fold increase in the maximum peak height and peak width for the specimen interrupted after a life-fraction of 40% compared to the values at the life-fraction of 10%. This increase in surface roughness is attributed to the formation of new slip markings which evidently is the outcome of the strain burst.

Surface morphology for the specimen cycled to 70% of life-fraction is presented in Fig. 1(e) (i) (SEM image) and 1(e)(ii) (AFM image). The SEM image shows the presence of deeper cracks compared to Fig. 1(d) indicating the occurrence of extensive crack nucleation from the PSMs during the period. The AFM image showed similar *peak valley* surface morphology as observed in Fig. 1(c) and (d) with no detectable change in terms of maximum peak height (~200 nm) or peak width (~0.5 μm) (Fig. 1d(iii)). This may be accounted for, by the absence of any major strain burst occurring in this period (40–70% of life-fraction) (Fig.1a). The creep strain accumulating in this period only gets localized in the in the