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# Disappearance and reappearance of serrated plastic flow under cyclic loading: A study of dislocation substructures

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ABSTRACT

Serrated plastic flow was observed in cyclic stress–strain hysteresis loops both in tensile and in compressive components during strain controlled low cycle fatigue of annealed Zircaloy-2 at a strain rate of $10^{-4}\,s^{-1}$ and different strain amplitudes, at room temperature. Although there were only D-type serrations at higher strain amplitudes ($> \pm 0.75\%$) there were mixed serrations of D, A, B and C type at lower strain amplitudes ($\le \pm 0.75\%$). Depending on the cyclic stress response, transitions in dislocation structure from dislocation pile-ups to cell structure and cells to Corduroy structure were observed in the region of softening and secondary hardening. Disappearance and reappearance of serrated flow in the hysteresis loops were observed with number of cycles and it was proposed to be associated with transition in dislocation structure from planar to cell walls and finally from cell wall to Corduroy structure.

## 1. Introduction

Recently, occurrence of dynamic strain ageing (DSA) in annealed Zircaloy-2 under constant strain cycling at a strain rate of $10^{-4}\,s^{-1}$ at room temperature (RT) was established, for the first time, by the authors [1]. Rao et al. [2] reported the occurrence of DSA in the superalloy 718 under strain controlled fatigue at room temperature. They observed serrated flow in cyclic stress–strain hysteresis loops, predominantly of type C, both in solution annealed and in double aged conditions and attributed it to the repeated development of pile-ups and reduction in stress ahead of the pile-ups associated with planar slip deformation. DSA has been observed in various materials like superalloys, austenitic stainless steels, SA508Cl.3 forging steel, duplex stainless steel (DIN 1.4460), titanium and titanium alloys, magnesium alloys, and zirconium alloys under cyclic deformation [3–11]. In general DSA is a diffusion controlled phenomenon and occurs at intermediate temperatures ($0.3$–$0.6T_m$) arising from periodical pinning and unpinning of dislocations by solute atmospheres around the core of dislocations.

There are different manifestations of DSA under cyclic straining such as (i) serrations in stress–strain hysteresis loops, (ii) increase in number of cycles to attain $(\Delta\sigma/2)_{\max}$ with decrease in strain rate, (iii) decrease in plastic strain range with increase in temperature or decrease in strain rate for a given total strain range, (iv) increase in stress response with decrease in strain rate (negative strain rate sensitivity) or increase in temperature, and (v) large normalized cyclic hardening $(\Delta\sigma/2)_{\max}/(\Delta\sigma/2)_1$, where $(\Delta\sigma/2)_1$ is tensile stress amplitude in the first cycle and $(\Delta\sigma/2)_{\max}$ is maximum tensile stress amplitude, the ratio of which increases with decreasing strain rate [12].

DSA in zirconium and its alloys has been reported under uniaxial loading from occurrence of peak in yield stress–temperature plot [13–17], elongation minima [13,16,17], strain rate sensitivity minima [13,16,17], and activation volume peak [13,14,18] in the temperature range 250–450 °C. Most of the authors have attributed pinning of dislocations by oxygen. Some authors have reported two peaks in flow stress–temperature plot [16], strain rate sensitivity [13,16] and activation volume peak [13] corresponding to low temperature (300 K) and high temperature (675 K). Ramachandran and Reed-Hill [16] have attributed the low temperature peak to hydrogen atoms and the high temperature peak to oxygen, nitrogen, or carbon atoms. Similarly Yi et al. [13] observed a second peak at a higher temperature of 723 K

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(450 °C) and attributed it to iron atoms and the lower temperature peak between 573 and 673 K (300–400 °C) to oxygen atoms.

The objective of this paper is to characterize the deformation substructure in the Zircaloy-2 resulting from DSA during cyclic straining at room temperature and propose mechanisms for the appearance/disappearance of serrations.

## 2. Material and methods

Zircaloy-2, the material of the present investigation, was supplied by M/s. Nuclear Fuel Complex, Department of Atomic Energy, Hyderabad, in the form of rods of 14 mm diameter, processed by extruding billet of 150 mm diameter to 24 mm rod, followed by swaging and vacuum annealing at 730 °C for 3 h.The chemical composition of the alloy is presented in Table 1. Axial push–pull LCF tests were conducted under fully reversed strain cycle of sinusoidal wave form, at room temperature, using a 50 kN Servo hydraulic MTS™ (Model 810), at different total strain amplitudes ($\Delta\varepsilon_t/2$) of ±0.50%, ±0.60%, ±0.75%, ±1.00% and ±1.25%, at a strain rate of $10^{-4}\ \mathrm{s}^{-1}$. Cylindrical LCF test specimens with gage length and gage diameter of 15 mm and 5.5 mm, respectively, shoulder radius of 25 mm, and threaded ends of 12 mm diameter and 30 mm length on each side, were used. The gage section was polished with emery papers, up to the grade of 4/0, and finally with paste of alumina powder with water. TEM foils were prepared in an electrolyte containing 60 ml $HClO_4$, 240 ml n-butanol and 600 ml methanol at 35 °C and 20 V, using a Tenupol 5 (Struers) twin jet polisher.

## 3. Results

### 3.1. Stress response

The cyclic stress response of annealed Zircaloy-2 under strain-controlled cycling at room temperature appears to be quite complex and strongly dependent on strain amplitude [1]. Cyclic stress response of annealed Zircaloy-2 at a strain rate of $10^{-4}\ \mathrm{s}^{-1}$ with different regions of cyclic softening and hardening at low and high strain amplitudes, respectively, at room temperature, is shown in Fig. 1. Irrespective of strain amplitude there was also occurrence of a secondary hardening region at the later stage of cycling. The cyclic stress response was associated with serrated plastic flow at decreasing strain rate [1].

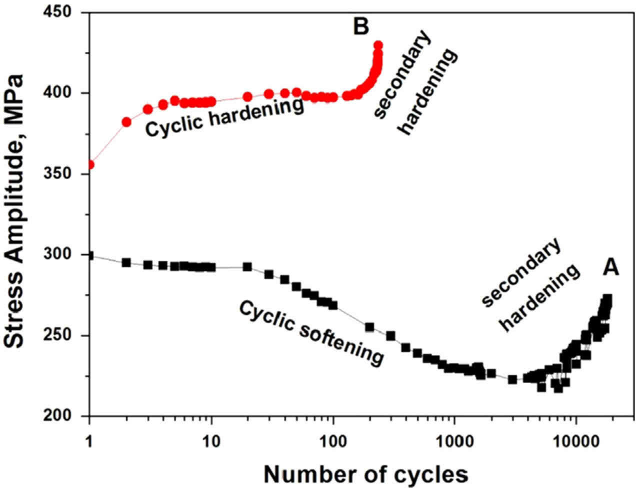

**Fig. 1.** Cyclic stress response of annealed Zircaloy-2 at strain rate $10^{-4}\ \mathrm{s}^{-1}$ showing different regions: (A) low strain amplitudes: ±0.50% and (B) high strain amplitudes: ±1.25%.

### 3.2. Serrated plastic flow

Serrated plastic flow both in tensile and in compressive parts of cyclic stress–strain hysteresis loops at total strain amplitudes of ±0.50%, ±0.75% and ±1.00% is shown in Fig. 2(a-d) respectively. The nature, yield drop/rise, and density of serrations were strong function of the strain amplitude and number of cycles. At higher strain amplitudes ($\geq \pm 1.0\%$) the serrations were predominantly of type D whereas at lower strain amplitudes ($\leq \pm 0.75\%$) there were mixed types of serrations (D, A, B, and C). The deformation started with the appearance of type D serrations during the initial cycles and changed to type A followed by A+B and subsequently by A+B+C type mixed serrations with number of cycles, at lowers strain amplitudes. Serrations of B type were prominently observed before the change of strain loading direction from tension to compression and vice versa. The occurrence of different types of serrations and their characteristics are reported in the review by Rodriguez [19]. The magnitude of rise/drop of stress and the density of the serrations increased with number of cycles. This was observed in the region of secondary cyclic hardening. Also the critical strain for serrated flow was found to decrease with the number of cycles at all the strain amplitudes. There was also a stage of partial or complete disappearance of serrations during cyclic loading in the region of secondary hardening followed by its reappearance after few cycles and remaining stable towards the end of fatigue life. This observation suggests that constant strain cycling of Zircaloy-2 at room temperature resulted in stress response similar to that of temperature effect on DSA.

**Table 1**
Chemical composition of the Zircaloy-2.

| Elements | Sn | Cr | Fe | Ni | Hf[a] | O[a] | C[a] | N[a] | H[a] |
|---|---|---|---|---|---|---|---|---|---|
| **Amount (wt%)** | 1.3 | 0.09 | 0.15 | 0.04 | <50 | 1040 | 64 | 40 | 8 |

[a] Amount is in ppm level by weight.

### 3.3. Disappearance and re-appearance of serrated flow

It was observed from critical examination of hysteresis loops of all the cycles at the lowest strain amplitude (±0.50%) that serrated flow almost disappeared after 5000 cycles and reappeared after 6000 cycles. Again after about 16,600 cycles the serrated flow almost disappeared in both tensile and compressive components of the strain cycle and started reappearing after 17,000 cycles. The disappearance and re-appearance of the serrations is shown in Fig. 3.

### 3.4. Dislocation structure

Figs. 4 and 5 show bright field TEM images along with the corresponding SAD patterns. The dislocation structures were quite different at low and high strain amplitudes. There was clear evidence of dislocation pile-ups in slip bands at the low strain amplitude of ±0.50% where there was pronounced serrated plastic flow in the stress–strain hysteresis loops. Fig. 4(a) shows pile-up of dislocations near the grain boundary in the slip bands. It may be seen that the inter-dislocation spacing was increased away from the boundary. Formation of fine dislocation loops may also be seen, as shown by arrows in Fig. 4(a). In addition to planar slip deformation there was also formation of dislocation cell network structure at a low strain amplitude of ±0.50% at the strain rate of $10^{-4}\ \mathrm{s}^{-1}$ Fig. 4(c). Planar dislocations are clearly seen along with the cell network structure. On the other hand at the