Microplasticity in Zr-2.5Nb alloy during thermal cycles

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Abstract: Stress relaxation by micro-plastic deformation was studied in polycrystalline Zr-2.5Nb alloy under very low stress 23,74 MPa during thermal cycling in the range 70 – 370°C. The described experiment belongs to creep and hydride cracking area of material science.

A dislocation motion, residual microscopic stresses and interactions of applied stress at small scale in grains are critical in understanding how the microstructure affects the macroscopic mechanical and physical properties of materials. The back stress is attributed to movable screw and edge dislocations impeded in their movement by structural defects and by interstitial impurities.

The obtained relaxation by micro-plastic deformation following pre-stressing to applied stress was logarithmic $\varepsilon = K \ln((t) + to)$, with the slope i.e. velocity proportional to the time. At higher number of cycles the relation between ε and t was like asymptote i.e. $d\varepsilon/dt$ approached to constant close to zero value.

Keywords: Zr-2.5Nb; Zirconium alloy; microplasticity ; thermal cycling; strain velocity, alongation velocity.

1. Introduction

Zirconium alloys are based materials for pressure tube in nuclear reactors. They are also applied on uranium fuel shields. Both applications are connected with good mechanical, corrosion and physical properties. The last one is characterized with very low cross section for thermal neutrons.

Stress relaxation by micro-plastic deformation in polycrystalline alloys under very low stress during thermal cycling is important area of creep. The applied stress values lesser then 10% of the macro-yield level and thermal cycling condition can demonstrate real exploitation conditions particularly when delayed hydride cracking appears [1,5,7]. An industrial training and simulations of shut down of power plants can be characterize with this type of experiments. Effective plastic strains are very low in the range $\varepsilon = 10^{-3}$ and therefore some technical difficulties must be overcome in monitoring such small strain. The 10 times mechanical magnification and optical system for monitoring plastic elongation was designed and elaborated [6].

In spite of small plastic strain due to very small-applied stress it may be important in many machines and devices in technology and in exploitation. Majority of machines work under cyclic stresses where stress limit as fatigue stress is fraction usually in between 0.2-0.6 of yield point. Even so, such small stress causes gradually structural and microstructural changes, which finally causes crack nucleation in the material. The microplastic deformation is particularly important in connection between machine elements due to established technical fit where several micrometers can change type of fit and important interactions between elements.

Due to importance of microplasticity phenomenon some focus on its structural mechanisms is investigated. Among others the Bishop-Hil and uniform lower-bound models and dislocation thermally activated cross-slip are used in understanding of microplasticity structural mechanisms [2,3]. A field of tensile stress can enhance diffusion and in case of interstitial hydrogen atoms some nucleation of hydrides can appear and following their cracking gives plastic deformation due to delayed hydride cracking phenomena (DHC) [1]. Dislocation slips, twining, crystal lattice defects, diffusion and stress induced phase transformation are the primary carriers of plastic deformation for crystalline materials. A dislocation motion,

residual microscopic stresses and interactions of applied stress at small scale in grains are critical in understanding how the microstructure affects the macroscopic mechanical and physical properties of materials. The back stress is attributed to movable screw and edge dislocations impeded in their movement by structural defects and by interstitial impurities. The structural relaxation mechanisms in the low-stress region are responsible for the small amount of cumulative plastic strain versus stress and time.

The micro-plastic deformation following pre-stressing of zirconium alloy Zr-2.5Nb was measured and described with mathematical functions for applied stress and thermal loading.

2. Sample preparation and experimental procedure

The samples made of Zr-2.5Nb alloy were prepared. The sample's working length was 25 mm and dimension of cross section area was 4.20x4 mm. The sample with holder (Fig.1) was placed in vertical electrical and programmable furnace. Special optical system was developed for monitoring and measuring length of the sample [6]. Temperature was measured directly on samples with NiCr-NiAl thermocouple with accuracy +-0.3 deg. Elongation of sample was measured with mechanical and optical laser system with accuracy +-0.2 μm [6].

Advanced X-ray diffraction examinations were carried out on original and used sample. After thermal cycling of sample the X-ray examinations under the same conditions were repeated in nondestructive way. The samples were under load equal to σ=23,74 MPa and continuously heated and cooled during thermal cycles within temperature range of 70-370°C. This thermal range of cycling was established due to real working temperature and to many experimental data about DHC [1,7]. Time period of a single cycle was 160 min with total cycles number equal to 100. In this paper the characterization of micorplasticity for examined alloy Zr-2.5Nb is described and considered due to meaning, mechanisms and application of empirical mathematical functions to describe obtained results.

The Figure 1 presents the schematics of sample and its holder.

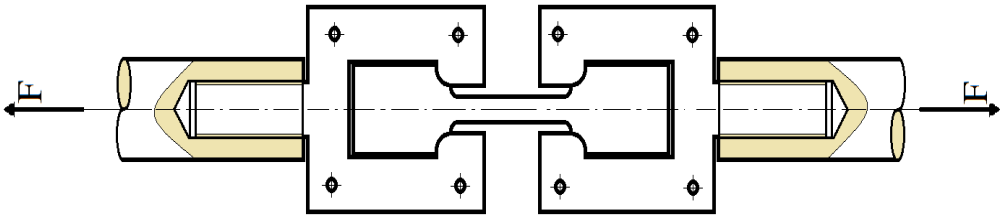

Fig. 1: The sample with holder schematics, which are inside the heat chamber of furnace.

3. Results and conclusions

Applied stress was a small fraction of yield point of this alloy i.e. 2.5%.

Two X-ray diffraction techniques were applied i.e. symmetric Bragg-Brentano (BB) and grazing incidence angle geometry. Both can characterize phase composition, state of structure and the second one can be used for residual stresses measurement [9]. Results obtained from X-ray diffraction patterns (Fig.2 and 3) show qualitative and quantitative differences between microstructure of the samples before and after the cycling experiment. The non-treated sample before experimental procedure consisted of zirconium α (hexagonal, P63/mmc, R: 03-065-3366) Sample after thermal cycling treatment consist of the same αZr as well as surface layer of zirconium dioxide ZrO_2 (Monoclinic, P21/c, R: 00-013-0307).

Illustration of results from loading and thermal cyclic experiments is shown on figure 4. As the example of strain and plastic deformation velocity of Zr-2.5Nb alloy sample under tensile stress 23,74 MPa during thermal cycling in the range of 70-370°C are presented on figure 5 - 7). Results on figures 5 and 6 can be described with logarithmic equations as:

$$\Delta L\ (t) = 9{,}6091\ln(t) + 50{,}876 \quad (1)$$

$$\varepsilon\ (t) = 0{,}0004\ln(t) + 0{,}002 \quad (2)$$

Where: ΔL – increment of length [μm], ε – plastic strain and t – time [min]

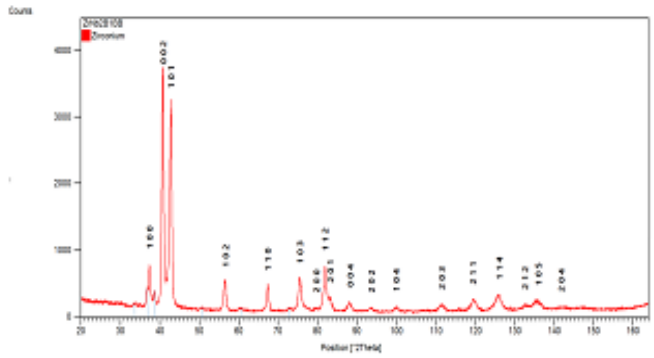

Fig.2. Diffraction pattern of Zr-2.5Nb raw sample before thermal cycling.

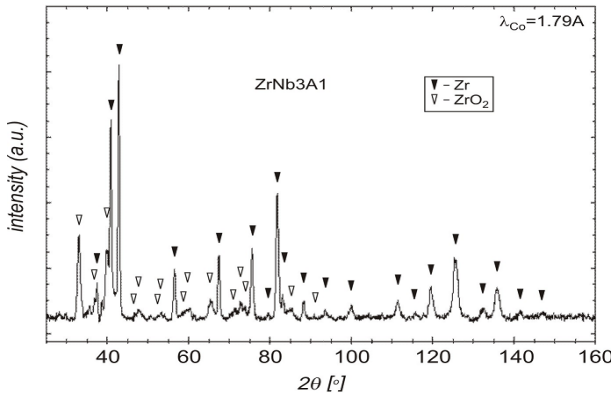

Fig.3. Diffraction pattern of Zr-2.5Nb sample after thermal cycling under 23,74 MPa tensile stress.

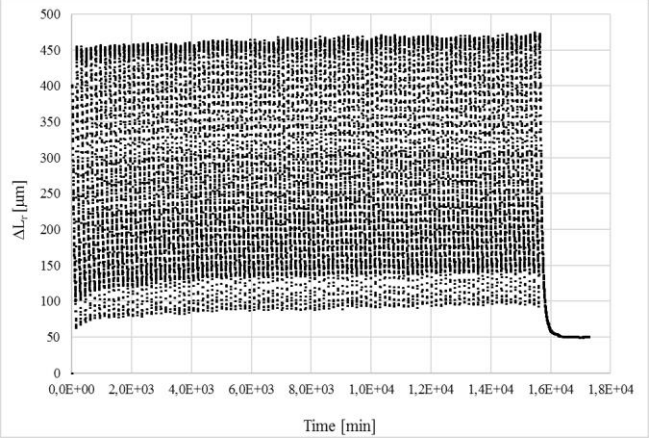

Fig. 4: Full experimantal data i.e. relation between sample's elongation and time for Zr-2.5Nb sample under tensile stress 23,74 MPa (T_{cycles}=70-370°C) .

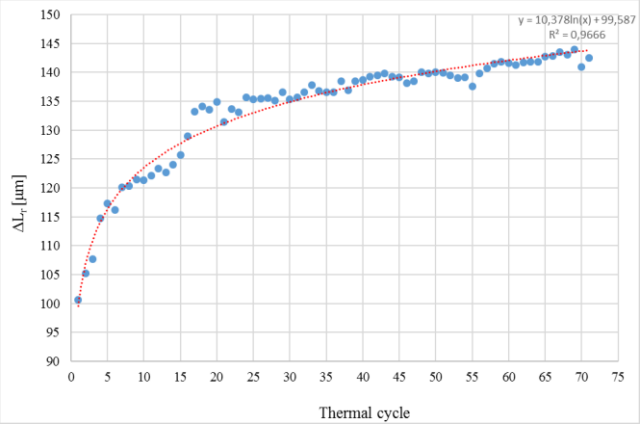

Fig. 5: The relation between elongation and time for Zr-2.5Nb sample under tensile stress 23,74 MPa at T_{cycle}=70°C with fitted log-normal distribution.

The elongation and elongation velocity of specimen at the beginning of experiment were established as $\Delta L = 1{,}01*10^{-4}$ m and $\Delta L/t = 5.36*10^{-9}$ m/s. The same data referring to the end of experimental cycling were following: $\Delta L = 1.41*10^{-4}$ m and $\Delta L/t = 1.6*10^{-10}$ m/s. These data recalculated to strain and strain rate for the beginning were: $\varepsilon=3{,}79*10^{-3}$ and $\varepsilon'=3{,}99*10^{-8}$ s^{-1} and after 100 cycles: $\varepsilon=5{,}46*10^{-3}$, $\varepsilon'=4{,}22*10^{-10}$ s^{-1}.

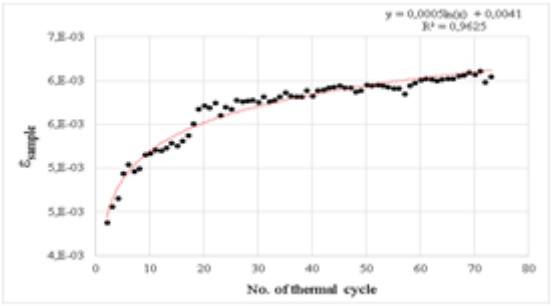

Fig. 6: The relation between strain and cycle number (time) for Zr-2.5Nb sample under tensile stress 23,74 MPa (T_{cycle}=70°C) with log-normal distribution.

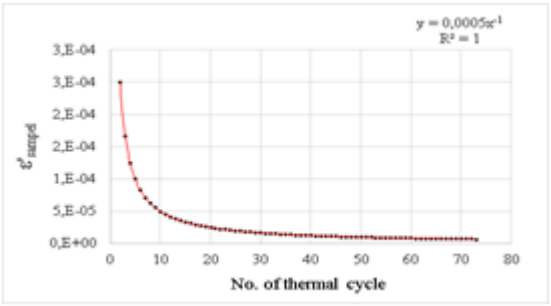

Fig. 7: The relation between strain rate and cycle number (time) for Zr-2.5Nb sample under tensile stress 23,74 MPa (T_{cycle}=70°C) with fitted function.

4. Conclusion

The obtained experimental results confirmed microplastic deformation of sample made of Zr-2.5Nb alloy under small tensile stress 23,74 MPa i.e. the microplasticity occurrence. Zr-2.5Nb alloy under very low tensile stress i.e. 23,74 MPa during thermal cycling in spite plastic plastic deformation thine coating of ZrO_2 oxide was gradually growing. Increase of plastic deformation versus time (number of cycles) was a log-normal distribution. The strain and strain rate decreases approaching to very low and almost constant value versus time.

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