Experimental Investigations of Anomalous Energy Absorption in Nanocrystalline Titanium under Cyclic Loading Conditions

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Abstract—An infrared thermographic scanning technique has been used to study the energy absorption and dissipation under cyclic loading conditions in the nanostructural bulk titanium obtained by severe plastic deformation of an initial coarse-grained material. It is established that the character of energy absorption in the nanocrystalline material exhibits qualitative changes, which are related to specific features of interactions in the ensemble of grain-boundary defects. The mechanisms of energy absorption are explained within the framework of notions of a new class of critical phenomena in mesoscopic systems with defects featuring structure-scaling transitions.

We have studied the process of energy absorption and dissipation in submicron-grained titanium under cyclic deformation conditions using a rapid method of fatigue evaluation based on the infrared thermographic scanning of samples [1]. The idea of our approach consists in establishing correlations between the level of energy dissipation and the onset of the final stage of fatigue fracture, which allows the fatigue limit to be determined at various levels of the average applied stress.

The experiments have been performed on polycrystalline Grade 2 titanium in the coarse-grained (microcrystalline) and submicron-grained (nanocrystalline) states. The nanostructural material was obtained by a severe plastic deformation (SPD) method [2] and had an average grain size of ~150 nm. Comparative data on the mechanical characteristics of Grade 2 titanium in the micro- and nanocrystalline states are presented in the table. Figure 1 shows the geometry and dimensions of test samples. The experiments were performed using an Amsler Vibrophore fatigue test machine, which provided uniaxial cyclic loading of samples at a preset level. The resonance frequency of samples was about 76 Hz.

The temperature field was monitored with a maximum image size of 320×240 pixels using a Jade III (Cedip Infrared Systems) near-IR camera operating in a 3–5 μm wavelength range at a sensitivity of no less than 25 mK at 300 K. The fatigue loading schedule consisted of periodic 30000-cycle blocks with a cycle asymmetry of 0.1. In every next block, the average applied stress was increased by 10 MPa. An increase in the average samples temperature was

Comparative data on the mechanical characteristics of Grade 2 titanium in the micro- and nanocrystalline states

State of material	Ultimate tensile strength σ_u, MPa	Conventional yield strength $\sigma_{0.2}$, MPa	Relative elongation at break δ, %
Initial coarse-grained (grain size, ~25 μm)	440	370	38
SPD + warm rolling (grain size, ~0.3 μm)	1090 ± 20	980 ± 20	13 ± 1

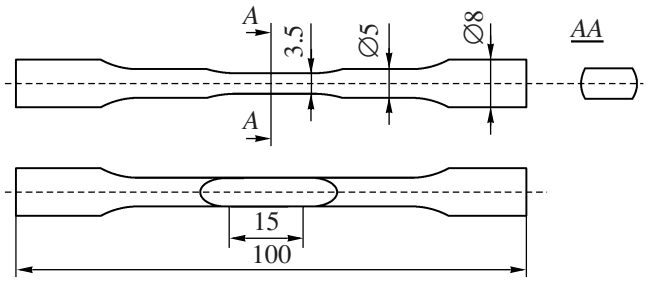

Fig. 1. Geometry of samples used for cyclic fatigue testing (working part, $15 \times 5 \times 3.5$ mm).

measured at each loading step. Between the blocks, the sample was unloaded and allowed to attain the state of thermal equilibrium with the ambient medium.

Figure 2 presents the results of experiments with titanium samples in the micro- and nanocrystalline state. As can be seen, the sample with micron-sized grains exhibits a nonlinear two-stage growth in the dissipated energy. According to the applied experimental method, the point of intersection of the approximating (dashed) straight lines in Fig. 2 provides an estimate for the fatigue limit (average stress, 80 MPa; maximum stress, 145 MPa) of coarse-grained titanium. The experiment was terminated when the sample temperature was outside the working range (75°C) of the camera operating at a 1100 μs exposure.

The results of IR scanning showed that the cyclic loading of nanocrystalline titanium is accompanied by qualitative changes in the character of energy absorption. At a small stress amplitude, the average temperature of the fine-grained sample only slightly exceeds that of the coarse-grained sample. When the applied stress reaches a level of the fatigue limit, the pattern qualitatively changes and the temperature increment in the nanocrystalline sample becomes significantly lower compared to that in the coarse-grained sample. A stable linear dependence of the temperature growth rate on the average stress is characteristic of the entire loading pattern for nanocrystalline samples. The sample temperature exhibits stabilization upon approximately 20 000 cycles, which reflects the ability of the nanostructural material to form an equilibrium system of grain-boundary defects and confirms the well-known theoretical results on the formation of a lattice of grain-boundary defects with the characteristic size (dislocation density) homogeneously increasing with the average applied strength over the entire sample volume.

The fracture of nanocrystalline samples upon cyclic loading had a quasi-brittle character and took place at a stress amplitude that was 35% (sample 1) and 40%

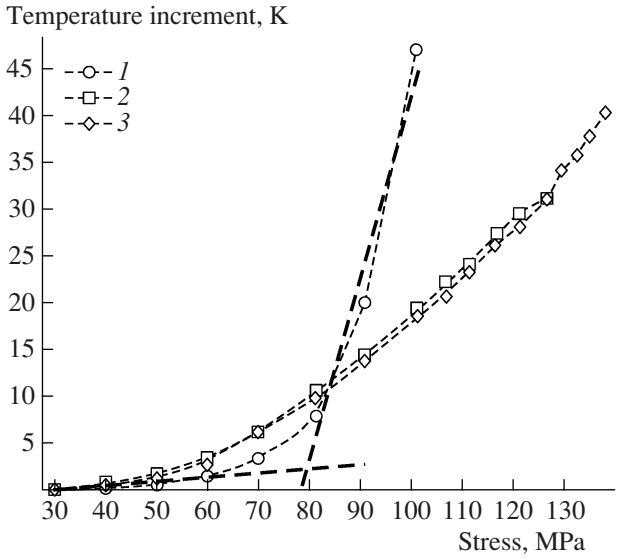

Fig. 2. Plots of the average temperature increment versus average applied stress for (*1*) coarse-grained and (*2*, *3*) nanocrystalline titanium under cyclic loading conditions. The dashed lines show linear approximations of the data for two stages of energy dissipation in the coarse-grained sample.

(sample 2) greater than that observed for titanium in the initial coarse-grained state.

The SPD treatment of titanium leads to the formation of ultradisperse nonequilibrium structures characterized by a high density of lattice and grain-boundary defects, which form long-range fields of elastic stresses [3]. For this reason, the nanostructural state of the material can be considered as metastable. As a result, the plastic and cyclic deformation of samples in this state is accompanied by anomalous absorption and dissipation of the deformation energy. Based on the statistical-thermodynamic theory developed previously for solids with mesoscopic defects, a new class of critical phenomena was considered, which are related to a collective behavior of the ensembles of dislocations (structure-scaling transitions) [4] and can explain the laws of transitions from the micro- to nanocrystalline bulk material. A specific feature of this class of critical phenomena (typical of nonequilibrium systems with structural defects) is the existence of an additional order parameter, called the structure scaling order parameter, which depends on the scaling characteristics of the medium and the interaction between defects. Based on the statistical description, the proposed thermodynamics and phenomenology presented a generalization of the Ginzburg–Landau approach and allowed qualitative differences to be established between the structure-scaling transitions in the corresponding regions of the new order parameter. These differences are related to the types of collective modes of the ensembles of defects characteristic of the quasi-brittle and viscous (plastic) bulk nanocrystalline states. The kinetics of the

structure scaling order parameter determines the relaxation behavior of a given material during the plastic shear, the features of transitions from the microscopic damage to macroscopic fracture, and the laws of energy absorption and dissipation in the course of deformation.

Qualitative differences in the behavior of materials in the micro- and nanocrystalline states are related to the collective modes of various types in dislocation substructures (ensembles of grain-boundary defects) formed under the conditions of structure-scaling transitions. These distinctive features are clearly pronounced in the course of cyclic loading—in particular, widely used cyclic fatigue testing, which is characterized by multiply repeated ($>10^6$ cycles) periodic variation of the amplitude of applied stress relative to a certain average level.

The kinetics of structure-scaling transitions is determined by the type of collective modes in the ensembles of defects, the change of which (accompanied by a sharp change in the symmetry of the system) leads to qualitatively different mechanisms of deformation. It was established [4] that, on the passage from microcrystalline to bulk nanocrystalline state, collective modes of the autosoliton nature change to spatially localized immobile structures formed on certain characteristic scales. This qualitative change in the type of collective modes is related to a change in the mechanism of deformation, which is characterized by a threshold yield stress for the autosoliton modes of localized plasticity in a microcrystalline state and by the formation of lattices of grain-boundary defects (dislocation crystals) on the passage to the submicrocrystalline material.

As is known, the energy balance in plastic deformation is characterized by the dissipation and configuration contributions. The former leads to an increase in the sample temperature, while the latter is determined by the amount of bound energy, which is contained in the fields of structural stresses of dislocation substructures on various scaling levels. For microcrystalline materials, the plastic deformation is realized by a sequential scaling structure-transition to dislocation substructures with increasing scales, until the shear mobility of these substructures would be exhausted and they become the foci of fracture. In a material possessing a nanocrystalline bulk structure, the scaling transitions are not as pronounced and the deformation is accompanied by the formation of a lattice of grain-boundary defects exhibiting relatively homogeneous growth. In this case, the onset of fracture is manifested by the formation of a cluster of critical grain-boundary defects that is accompanied by the development of events according to a quasi-brittle scenario. This difference in the evolution of dislocation substructure in micro- and nanocrystalline materials is analogous to that involved in the first-order phase transitions.

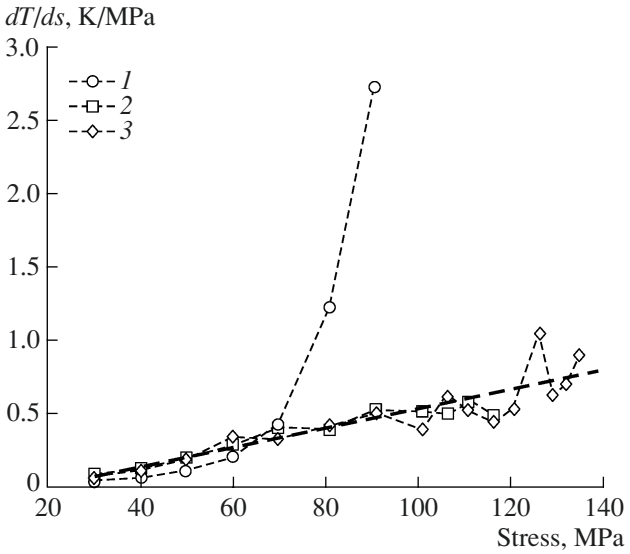

Fig. 3. Plots of the average equilibrium temperature growth rate versus average applied stress for (*1*) coarse-grained and (*2*, *3*) nanocrystalline titanium under cyclic loading conditions. The dashed line shows a linear approximation of the data for nanocrystalline titanium samples.

The above conclusion concerning the qualitative difference between mechanisms of the energy dissipation and the fracture of micro- and nanocrystalline titanium is confirmed by an analysis of data presented in Figs. 2 and 3. As can be seen from Fig. 3, the dependence of the rate of energy dissipation on the average applied stress is well approximated by the linear function for both samples of nanocrystalline titanium, which is qualitatively different from the behavior of the coarse-grained material. For the fine-grained material, the fatigue limit cannot be treated as a threshold stress leading to a qualitative change in the mechanism of structural relaxation (possessing pronounced multiscale character in the final stage of deformation). An increase in the average temperature of the nanocrystalline sample (and, hence in the total power of heat sources appearing die to the evolution in the material structure) is directly proportional to the energy (squared stress amplitude) spent for deforming the same at all values of the applied stress. This fact characterizes the ability of a submicrocrystalline material to effectively realize the structural (configuration) channel of energy absorption, thus involving the entire nanocrystalline material bulk into this process. The less significant difference in the breaking stress between the microcrystalline and nanocrystalline materials in the course of cyclic deformation (as compared to the case of quasi-static loading with evaluation of the ultimate tensile and yield strength of coarse-and fine-grained materials) is related to specific features of the cyclic loading and the corresponding kinetics of defect accumulation. The structure relaxation as such is

less pronounced in fine-grained materials than in coarse-grained (plastic) ones, which is related to the effects of deformation localization. This factor also accounts for the quasi-brittle character of fracture observed in nanocrystalline materials, where a local cluster of bound grain-boundary defects is formed. In fine-grained materials, in contrast to coarse-grained ones, the passage to the final stage of fracture is not accompanied by the localization of deformation and the related structural relaxation.

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REFERENCES

1. M. P. Luong, Nucl. Eng. Design **158**, 363 (1995).
2. V. Latysh, I. Semenova, G. Salimgareeva, et al., Magter. Sci. Forum. **503–505**, 763 (2006).
3. R. Z. Valiev and I. V. Aleksandrov, *Nanostructural Materials Obtained by Plastic Deformation* (Logos, Moscow, 2000) [in Russian].
4. O. B. Naimark, Fiz. Met. Metalloved. **84** (4), 5 (1997) [Phys. Met. Metallogr. **84**, 327 (1997)].