Effects of nano and submicrocrystalline states under a wide range of loads

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Abstract. The scale in nanomechanics ($10^{-10}$-$10^{-9}$ m) has a fundamental physical meaning and can be considered as a control parameter when considering qualitative changes in the properties of polycrystalline materials when the grain structure changes in a wide range of load intensities. The existence of this parameter can lead to effects beyond the scale, which are traditionally considered in mechanics, and are accompanied by the influence of quantum effects on the deformation properties and failure of materials. New ideas about submicrocrystalline and nano-scale structure of materials achieved by the use of high-resolution methods (synchrotron tomography, soft X-rays, nanoindentation) have led to qualitative changes in approaches to the study and description of the properties of materials, including biomaterials. This may provide new information on the processes of deformation and fracture at the micro- and nano-scale level and lead to new approaches in the design and evaluation of the reliability.

1. Introduction

New ideas about nanoscale structure of materials achieved using high-resolution methods (e.g., synchrotron tomography, soft x-ray radiation, nanoindentation) have led to qualitative changes in approaches to study and describe the properties of materials, including biomaterials. Being an intensively developing area with promising applications, research has stimulated a number of transdisciplinary directions in modeling, such as mechano-thermochemistry, mechanobiology, which suggests the development of new principles of modeling. As applied to materials science, this can provide new information on the processes of deformation and fracture of materials at the micro- and nanoscale levels and lead to new approaches in the design and assessment of the reliability of construction in aviation, space, power energy techniques. Characteristic scale was introduced in [1] for crystalline solid

$$\xi = \left( \frac{h}{\rho (G / \rho)^{1/2}} \right)^{1/4} = \left( \frac{h}{C \rho} \right)^{1/4} \sim (10^{-9} - 10^{-10}) m$$

which includes the Planck constant $h$ and the classical parameters of the medium: density $\rho$, elastic modulus $G$, and longitudinal elastic waves velocity $C$. The ratio of $\xi$ and the interatomic distance $r_0$ for various crystalline materials gives $\alpha = r_0 / \xi \sim 2.4 - 5.0$. The scale $\xi$ can be considered as the length determined by both quantum and classical (mechanical) effects.
The failure problem demonstrates a number of statements for which understanding of the structure evolution at the nanostructural level is crucial, since it naturally relates effects on macroscopic, meso-, nano- and quantum scales. In this case, one of the key questions is what quantum effects can extend to other large-scale levels. An example is the correspondence of quantum dots in optoelectronics to their sizes, the critical length of which is close to 10 nm. The illustration of these links is the correlations between the laws of fractoluminescence and the stages of failure [3], which is an illustration of the critical behavior of systems at the quantum and microscopic levels with the corresponding scaling laws.

Descriptions of the relationship between atomic structures and mechanical behavior (plasticity, failure) were developed in the works of R. Peierls, F. Nabarro, V.V. Novozhilov, D. Dugdale, G.I. Barenblatt and made it possible to substantiate approaches that take into account the formation of cohesive zones and effects non-locality, as well as to explain the multiscale effects of plastic deformation and fracture under very high cycle fatigue and intense loads.

2. Critical submicrocrystalline scales in fracture and plastic deformation

Critical lengths correspond to qualitative changes in the behavior of materials, such as violation of the Hall-Petch law upon transition to the nanocrystalline state [2], stages of damage-failure transition in very high cycle fatigue [3], initiation of localized (adiabatic) shear bands under dynamic loading [4], the structure of cohesive zones, the formation of failure waves under shock-wave loading of brittle materials [5]. The formation of a nano- and submicrocrystalline structure under deformation has characteristic signs of a critical phenomenon and is associated in [6] with non-equilibrium (structural-scaling) transitions in ensembles of mesodefects, which result in multiscale fragmentation of the crystalline phase. The development of the fragmentation process and grain size distributions are determined by the intensity of the deformation process (plastic strain rate), which is localized on corresponding spatial scales. Bimodal grain size distribution at moderate strain rates, for example ECAP processing, is characteristic of the scenario of multiple plastic strain localization regions. Dynamic deformation, accompanied by the adiabatic shear banding, includes regions with crystalline fragments close to the nanostructured scale. The formation of these regions indicates the involvement of multiscale mechanisms of plastic relaxation. Features of plastic fragmentation as a critical phenomenon manifest themselves in the submicrocrystalline range of grain sizes in the form of anomalies in the mechanical energy absorption, that is typically of the first kind phase transitions [7].

The existence of a characteristic scale $\xi$ can be associated with a “damage quantum”, that is a scale at which relaxation mechanisms are “exhausted”. The signs of achieving a nanostructured scale in fragmentation processes correspond to a qualitative change in the distribution statistics of fragments and fractoluminescence signals (from bimodal to unimodal) during dynamic fracture of brittle materials. The statistical laws of grain fragmentation, corresponding to unimodal and bimodal distributions, also manifest themselves as morphological features of failure surfaces in Very High Cycle Fatigue (VHCF) [3], figure 1. The existence of a critical scale explains the self-similar laws of nucleation and growth of cracks under conditions of VHCF, are related to the ratio of characteristic scales (damage localization $L_c$, “cohesion zones” determining the growth kinetics of small and macroscopic cracks) $L_{pz}$, and scale $\xi$ : $\lambda_\xi = L_c/\xi > 1$ and $\lambda_{pz} = L_{pz}/\xi > 1$, which suggests the existence of self-similar intermediate asymptotics, respectively for the stage crack initiation (“fish-eye”) and crack propagation, figure 1.
3. Self-similar fragmentation laws under dynamic and shock-wave loading

The statistical features of the fragmentation dynamics were studied in in-situ experiment on dynamic loading of fused quartz rod with recording fractoluminescence associated with fracture zone formation. By analyzing spatiotemporal distributions (fragment sizes, time intervals of fracture luminescence signals), we found the power-law distribution that is typical of non-equilibrium systems revealing the self-organized criticality (figure 2). The power-law universality of the fragmentation statistical laws can be interpreted as the “degeneration” of spatial and temporal scales up to the macroscopic sample length. The study of load intensity and material structure on multiple site fracture in ceramics was based on analyzing fragmentation statistics data. Rod samples were dynamically compressed (the split Hopkinson-Kolsky pressure bar test [8]), and tubular samples were loaded by shock pulse [9].

- **Figure 1.** Fracture surface of the Ti6Al4V titanium alloy sample under VHCF ($10^9$ cycles) with characteristic fracture zones: 1 — “fish-eye” region of fracture initiation, 2 — propagation of “small cracks”, 3 — crack propagation according to the Paris kinetics [5].

- **Figure 2.** Cumulative distribution function of fractoluminescence time intervals (fused quartz sample).

- **Figure 3.** Specific quantity of fragments vs energy density: ♦ — quartz; ■ — granite; ● — ZrO$_2$; ▲ — Al$_2$O$_3$; O — SiC [9].
Figure 3 shows the dependence of the specific quantity of fragments on the impact load intensity (strain energy density). The processing of experimental data for five materials indicates that an increase in the number of fragments per unit mass \( N_m \) with the loading rate increase is described by the power law.

4. Failure waves as the limiting case of fragmentation

The limiting case of fragmentation is the initiation of “failure waves” in shocked fused quartz that has the nature of “delayed failure” and was explained as the “resonance” failure mode [5]. The “failure wave” was experimentally studied in a Taylor test [6] with high-speed framing of shadow images in a fused quartz rod after its collision with a rigid target at the velocity 534 m/s (figure 4).

![Figure 4](a)  
![Figure 4](b)

**Figure 4.** Failure wave in a fused quartz rod: (a) - high speed framing in the Taylor test; (b) \( X-t \) diagram 1 — compression wave, 2 — failure wave, 3 — collision surface.

The first shadow region corresponds to the compression wave travelling with the velocity \( V \approx 6.6 \) km/s. The second wave follows with constant lag with the velocity \( v_{fw} \approx 4 \) km/s, that is a wave front separating the solid and the fully comminuted material. It was shown in [10] that a failure wave propagates as “delayed” failure governed by “resonance” excitation of the collective blow-up modes of defects. Distributions close to unimodal should be observed during the formation of failure waves, as the limiting case of fragmentation with the formation of fragment sizes close to \( \xi \).

5. Structural investigation of localized plastic shear mechanisms and grain refining

The localized plastic shear effects at high strain rates and grain refining were studied for Al6061 and AlMg6 aluminum alloys, and steel during collision of a long rigid projectile with a plane target, in situ recording of temperature field dynamics (CEDIP Silver 450M infrared camera) and free surface velocity (the Doppler interferometry (VISAR)) on the back side of the target [4]. Multiscale structural relaxation mechanisms governed by the collective behavior of defect ensembles, plastic flow localization, and failure was investigated by structural analysis of the surface profile by a NewView-5010 interferometer-profilometer with subsequent calculation of the scale invariant (the Hurst exponent) and spatial scales of a region of correlated microshear behavior. The calculation of the scale invariants (structural scaling exponents) and corresponding scales allows us to find a relationship between shear localization stages with the signs of “criticality” due to correlated behavior of defects and the sizes of strain localization and adiabatic shear failure zones.

The recovered Al6061 targets after dynamic loading were examined by optical microscopy in the diameter cross-section after mechanical polishing and electrochemical etching (figure 5). Microetching for different resolutions revealed a sharp increase in the density of slip bands, which reaches a
critical density in the vicinity of the fracture surface. The sequence of coarse slip bands consisting of a system of 15–20 fine bands demonstrates spatial similarity, which is confirmed by the calculation of scale invariants. The fracture surface formation is accompanied by the formation of multiscale porosity and its clustering.

![Figure 5. Aluminum alloy microstructure near the area of “plug” formation (optical microscopy, ×500) (a); subsurface layer microstructure: coarse (b) and fine slip bands (c).](image)

Microstructural analysis by transmission electron microscopy has shown that the projectile penetration into the target causes the elongation of subgrains into bands and their fragmentation in a thin material layer (figure 6). One can clearly see dislocation pile-ups inside crystallites; the crystallite boundaries are smeared. The initial coarse-grained structure is refined into an ultrafine-grained one with the grain size ~ 300 nm from the initial 1.52 mcm.

![Figure 6. Structure of the deformed layer near the impact surface: electron microscopic (light field) image (a), microdiffraction pattern (b).](image)

![Figure 7. Electron microscopic image of the structure of highly deformed layer: light field (a), electron diffraction pattern (b), dark field (c).](image)
The micro-diffraction patterns of such a structure have a great number of reflection points (figure 7), bearing witness to the appearance of high-angle grain misorientations. It can thus be concluded that, along with low-angle boundaries, high-angle boundaries are formed within initial subgrains due to rotational modes that might be associated with the ordering of dislocation structures.

6. Results and discussion

The basic aspects of fragmentation refer to statistical and energetic regularities of non-equilibrium “critical systems”. Important features of the “critical behavior” are manifested in statistical fragmentation laws depending on the state, initial structure of the material, and load intensity when “narrow” (exponential) and “wide” (power-law) statistical distributions as well as their combinations are observed. The developed concepts of multiscale defect evolution as a critical phenomenon, such as structural scaling transitions, revealed a relationship between gauge (scale-invariant) properties of a “solid with defects” as a nonequilibrium system, self-similar solutions characterizing multiscale collective defect modes. The appearance of the defect modes defines the structural relaxation mechanisms (localized plasticity) and damage localization mechanisms with the “blow-up” mode kinetics that causes crack nucleation. The statistical fragment distribution in the material regions traversed by “failure waves” must reflect the maximum fragmentation level achieved within the acoustic range of energy density and approach monodisperse fragmentation with minimum particle size.

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