On the Limit of Metals Disintegration by Method of Mechanical Dispersion

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Abstract

Mechanisms restricting the possibility to increase dispersity of powders produced by mechanical grinding method have been considered. Transformation of nanocrystal powder particles into super-plastic state has been demonstrated to be the main mechanism, whereupon disintegration stops, and all further treatment amounts to change in the shape of particles. Through introduction the notion of macro-dislocation – the linear defect of irregularity in the nanocrystal grain packing – both the process of transformation into super-plastic state and the process of disruption are described. Estimations of disintegration limit for metal powders have been made. Results are in good agreement with experimental data obtained by atomic power microscopy method (APM) on the iron powder ground with surface-active substances (SAS) added. A sketch of formation and development of nanostructure and phase constitution of powder particles being ground in ball mills in liquid organic media has been suggested, which takes account of the process features connected with multiple high-velocity cold-hardening, high temperature polygonization and recrystallization, and multiple re-chill in liquid media.

INTRODUCTION

Solution of the problem to produce highly dispersed powder systems is an urgent task for many technological applications. However, for majority of currently known methods of powder production there are restrictions, which determine the maximum particle size, which makes further grinding impossible. There is evidence of attempts to connect this maximum size with various parameters of mechanical impact [1–3]. In this connection particular significance is taken on by analysis of dispersion methods, which make possible to vary parameters of treatment modes over a wide range. One of such methods is mechanoactivation (MA) in liquid organic media with surfactant (SAS) added [1, 4–7].

It has been known that adsorption of substances, which significantly decrease free surface energy of solids, leads to sharp increase in their grinding intensity. This phenomenon was discovered and studied in details yet in the first articles devoted to research of adsorptive decrease of rigidity [4, 5, 8]. Especially encouraging is an application of SAS in mechanical grinding of plastic metals, which cannot be ground in the absence of adsorption-active media. Thus, it was shown in [9] that, in the presence of organic surfactants or electrolyte solutions, grinding of copper, silver and other metals is feasible.

Purpose of the present investigation is the recognition of physicochemical mechanisms imposing restrictions on the possibility of grinding through MA method. Theoretical examination of possible mechanisms of plastic deformation and destruction of powder particles has been carried out. Data provided by atomic power microscopy (APM) and X-ray structural analysis (XSA) are consistent with model sketch as a whole.

MODEL SKETCH

Particularity of MA method is that powder particle experiences blow compression load according to upset sketch [10]. Period of action t is defined by mill rotation rate and is in the range $10^{-3}-10^{-6}$ s. Particles experience strong dynamic cold-hardening through intensive plastic deformation and get heated to high temperatures T (about melting point T_{melt}). At $T \pounds 0.4T_{melt}$ cold-hardening gives rise to cellular dislocation structure and consequent pre-recrystallization polygonization [12, 13]. On attainment of $T > 0.4T_{melt}$, the primary recrystallization starts, which centers emerge at multiple defect conglomerations [12, 13]. They cannot grow considerably in time t, since particle, upon impact termination, cools down in the ambient liquid media up to the temperatures close to room ones. So upon the first blow the structure of particle contains multiple isolated nanograins left behind from recrystallization centers, which have not had time to grow. Nonrecrystallized particle region contains the remains of polygonized structure. The subsequent blows will repeat the same mechanothermal impact. Since nanograins are not subject to plastic deformation (the reasons for this are considered below), they practically do not change in time t. Over the rest region of the particle the accumulation of the following nanograin generations will take place until the whole particle gets the nanocrystal structure. In addition, the particles will be saturated by admixtures, which are the wear products of grinding media [14]. Described here and presented in Fig. 1, a sketch of nanostructure formation in powders obtained by MA method is apparently of rather general character, and is applicable for many plastically deformable materials, in particular, for metals and alloys. This is related with the fact that only evidence widely known and reliably established from the theory and experience of mechanothermal treatment of metals [10, 12, 13, 15] together with features of MA method [6, 7] have been used for this sketch development. Let us also add to said above that grinding in the ball mills ranks among the methods for obtaining metals in nanocrystal (NC) state [16].
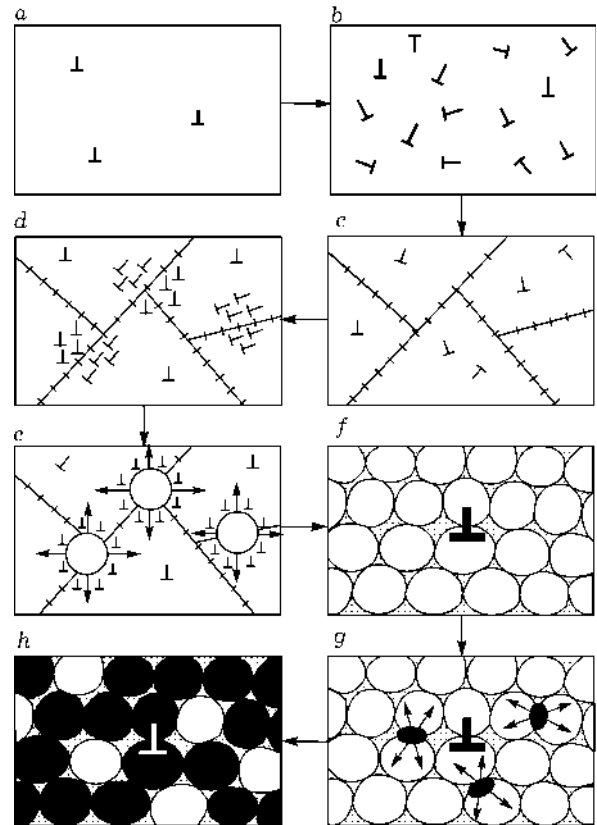

Fig. 1. Formation of nanostructure and phase constitution of particles of powders disintegrated in ball mills: a − initial defective structure of a crystallite of polycrystal powder particle (prior to grinding the structure is monophase); b − growth of consistence of dislocations at the beginning of grinding process; c − formation of crystallite substructure by polygonization at high temperatures; d − emergence of recrystallization centers in the form of microregions supersaturated with dislocations; e − formation and growth of isolated nanograins; f − completion of recrystallization processes, formation of nanostructure with mobile macrodislocations (symbol ^ marks the location site of the center of macrodislocation nucleus); g − supersaturation of nanostructure boundaries by admixtures and birth of and growth of grains of new phase; h − multiphase nanostructure after a long time grinding.

A trait of NC state is that the grain volume of nanocrystal is practically flawless [17, 18]. Dislocations in this case come out to the grain borders to form there amorphous-like thin-layer state of matter, having at high temperatures the properties of liquid phase [19, 20]. Actually, diffusion and self-diffusion coefficients along the dislocation pipes and at the inter-crystallite borders are closely matched to such ones in liquid melts [21], and yet the structure is very much disordered. That is relaxation period with respect to mechanical impact for such

media should be close to the ones of a liquid, *i. e.* the media at nanocrystal borders should show viscous liquid characteristics at strain. We mention that for lack of defects nanograins must be rigid [10, 12, 13] and their shape can conveniently be taken as globular, because only in this case the minimum of surface energy is achieved. Hence, the powder particles under MA conditions can be thought of as composite comprised of compactly packed indeformable globes sealed on the boundaries by liquid-like media.

Such material can be completely treated in terms of macrodislocations theory developed in [22] to account for plasticity of polycrystals with molten grain boundaries. The essence of the model is that in the materials under consideration the plastic deformation is effected not by the way of ordinary dislocations built of atoms, but by macrodislocations, which are the linear defects of disorder in regular packing of nanograins. This model is shown in Fig. 2. Were the nanocrystal the regular packing of nanograins, then it would appear at planar
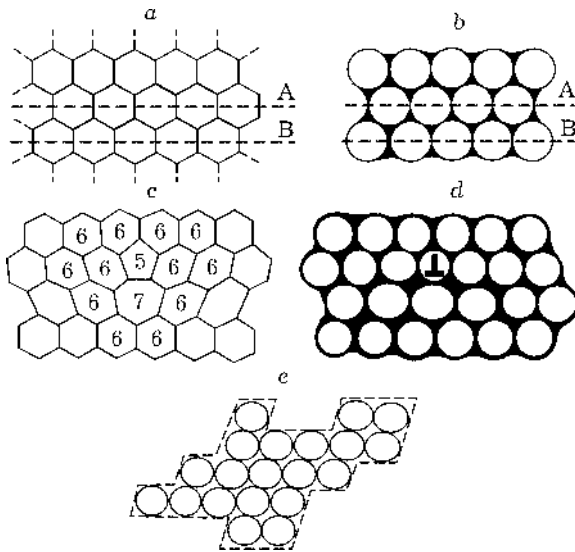

Fig. 2. Model of plastic deformation of nanostructure material: *a* — fragment of two-dimensional planar model of polycrystal with periodically structured arrangement of grains; *b* — fragment of two-dimensional crystal with loosened boundaries (arrangement structure of grains is periodic); *c* — the simplest topological defect of ideal hexahedron packing on plane (numbers indicate the quantity of polyhedron facets); *d* — structure obtained after loosening of boundaries of the polycrystal containing topological defect in grain packing shown in Fig. 2, *b*; *e* — steps derived from macrodislocation rise to the particle surface.

section so as it is shown in Fig. 2, *a*. Allocation of liquid-like media on its boundaries would give the sketch depicted in Fig 2, *b*. However, such structure is very firm, so a kind of model [22] modification has been carried out. The regular packing of nanograins contains defects of the type depicted in Fig. 2, *c*. Allocation of liquid-like media on the nanocrystal boundaries brings into existence a macrodislocation depicted in Fig. 2, *d*. Upon mechanical impact these macrodislocations will move and bring about plastic deformation by means of intergrain slipping. In [22] the conditions have been considered, under which plastic deformation of the material will be mostly determined by dislocation transfer process.

In the general case total deformation e under load for a considered composite matter with macro-dislocations is sum of three addends

$$\varepsilon = \varepsilon_{in} + \varepsilon_{\tau} + \varepsilon_{rot} \qquad (1)$$

where e_{in} is attainable interior deformation of grains-globes distorting their globular shape, e_{τ} is deformation through progressive shift of compactly packed lines of globes relative to each other, e_{rot} is deformation introduced by globes rotation.

Consider these addends individually with regard to the nanocrystall structure particularities. The value of e_{in} essentially depends on the globes friction level along the boundaries of one another. At high temperatures the liquid-like media on the inter-crystallite borders causes practically no resistance while slipping, if it occurs at velocities not so high. Therefore, the effect of friction at the surfaces can be neglected. We mention also that any distortion of the globe shape always tends to increase the surface energy of the system. For nanocrystal this increase may be big enough for reasons of large area of intercrystallite borders.

Considering in addition elevated rigidity of the nanograin body, the value e_{in} may be thought of as practically equal to zero under the circumstances involved. In this connection, contribution e_{rot} to the total deformation becomes equal to zero as well, so far as boundary-friction-free rotation of fixedly centered sphere does not deform the matter as a whole.

Hence, total plastic deformation under our circumstances is determined by e_{τ} addend,

which accounts for the deformation of inter-grain slipping in progressive moving of grains without rotation.

A simple method to estimate the value of e_t on a basis of macrodislocation model advanced in [22]. In its mechanical properties the nanocrystal comprises bi-phase composite elastic-and-viscous media, which behavior under load essentially depends on the deformation velocity. Description of such media properties can be substantially simplified with consideration of an equivalent to it in the long-wave limit, continuous isotropic media with shear modulus G, Poisson coefficient n and yield stress s_T. These characteristics are first to be calculated on the basis of specific features of nanocrystal structure. Within the limits of such media the theory of macrodislocation is no different from the theory of conventional dislocations [23].

With use of this, an estimation of yield stress for nanocrystal may be carried out in the framework of well-known model Peiers – Nabarro:

$$\sigma_T = \frac{2G}{1-v}\exp\left(-\frac{4\pi\xi}{b}\right) \tag{2}$$

where b is Burgers vector of macrodislocation ($b = 2R$), R is nanograin radius, $x \gg b$ is macro-dislocation width.

Maximum shift strains on the nucleus of macrodislocation do not depend on Burgers vector and are equal

$$\sigma_{\max} = \frac{G}{2\pi\left(1-v\right)} \tag{3}$$

Hence we find a condition whereupon the grains always hold the sphere shape and so the model of macrodislocations is acceptable for description of nanocrystal plastic deformation:

$$\sigma_{T,S} > \frac{G}{2\pi\left(1-v\right)} \tag{4}$$

where $s_{T,S}$ is the yield stress of the globe-nanograin matter.

The value of G is calculated in [22] for arbitrary polycrystal of sphere-shaped grains separated by wetting liquid phase. For nanocrystal account must be taken of the following features. The liquid-like media in this case takes the form of very thin layers, so it transfers between the globes not by conventional liquid flow, but by means of barodiffusion. Then with formulae [22] it is easily derived

$$G = \frac{1}{3}\left\{K_l\left[\frac{R}{V_l^2\left(R + 4\sqrt{\dfrac{D\Omega\delta K}{12kTRv_d}}\right)} - V_l\right] - K_s V_s\right\} \tag{5}$$

where K_l and K_s are compression modulus for liquid-like and solid phase correspondingly, V_l and V_s are fractions by volume of these phases, D is self-diffusion coefficient in the liquid phase, W is atomic volume, d is border width, k is Boltzmann constant, T is temperature, v_d is velocity of macrodislocation moving.

To perform the process of nanomaterial superplasticity it is essential that

$$G \leq 0 \tag{6}$$

Therewith the condition (4) is automatically complied with [22]. We note that this condition complied with may be insufficient. To effect the superplasticity mode it is necessary, in parallel with absence of resistance to macrodislocation movement in the bulk of particle, to provide also their unimpeded rise to outer surface. In reality any rise of macrodislocation from volume is connected with increase of the particle outer surface area, and hence there always exists some energy barrier impeding this process. Surfactants may essentially decrease this barrier, thus having a profound impact on the transformation to superplasticity mode. That circumstance is particularly important to take into consideration when viewing finely dispersed systems with well-developed outer surface. We mention as an adjunct that significant dispersion in nanograins size distribution may also suppress essentially the effect of superplasticity. Therefore condition (6) can be regarded as a criterion of superplasticity but for uniform nanostructures with no-barrier rise of macrodislocation to outer surface. Supposing these conditions are complied with, we estimate the radius of nanograins, for which the superplasticity effect will take place as applied to MA method. Substituting $d \gg 10^{-9}$, $W \gg 10^{-29}$ m^3, $kT \gg 10^{-19}$ J ($T \gg 10^3$ K), $D \gg 10^{-9}$ m^2 s^{-1}, $K_l \gg 10^{11}$ J m^{-3}, $K_l \gg K_s$, in (6), (5) gives a condition, which links R, V_l and v_d as

$$R \leq 10^{-5} V_l^{4/3} v_d^{-1/3} \qquad (7)$$

Assuming here V_l equal to boundary volume

$$V_l = 3\delta/2R \qquad (8)$$

gives

$$R \leq 10^{-7} v_d^{-1/7} \qquad (9)$$

Evidenced by estimation (9), the deformation velocity dependence is rather loose, and over velocity range

$$10^{-3} \mathrm{m\ s^{-1}} \pounds\ v_d\ \pounds 10^3\ \mathrm{m\ s^{-1}} \qquad (10)$$

superplasticity effected through macrodislocations moving in combination with mass transfer by diffusion along the boundaries, may be thought of as feasible when

$$R\ \pounds\ 10^{-7}\ \mathrm{m} \qquad (11)$$

Needless to say, the condition (11) may serve as criterion only for chemically pure nanomaterials.

If nanocrystal contains mobile surface-active admixtures, other relationships should be applied to estimate the value of R. However, on general grounds, one can reason that such admixtures may do nothing but increase the macrodislocation mobility and widen the allowable interval for parameter R value. It follows from the fact that mobile surface-active admixtures make greater contribution in mass transfer by diffusion than dominant metal atoms. Resulting effective coefficient of mass transfer by diffusion in formula (5) must grow, and hence value of G must decrease for the same values of R.

Despite the apparent obviousness of the effect, the accurate calculation of the G value in this case is complex to carry out. Closer examination of this issue deserves the special consideration; as for now, for present purposes, the compliance with condition (11) would be quite sufficient. From this condition follows that in proper deciding on surfactant, nanostructured powder particles can be superplastic. That is their grinding stops at once and all further treatment of the matter results in change of particle shapes at a constant volume. As a rule, from this should result the powders of disk-shaped particles with very low ratio of thickness to diameter. Macrodislocations rise to outer surface should result in formation of steps akin to the ones shown in Fig. 2, e in dotted line. An important point here is that the step height should be a multiple of the nanograin average size and this fact may be studied experimentally.

To define the particle sizes and shapes and also to study their surface microstructure the method of atomic power microscopy (APM) has been used. The APM method is unique in that it allows to reproduce 3-dimension (3D) object images under very high (nanometer) resolution.

EXPERIMENTAL

Samples were obtained by grinding of iron powder with particle size of <300 mm in the ball planetary mill in heptane media and surfactant added (0.3 mass % of oleic acid) over a period of 1 and 99 h. Every 3 h of treatment, SAS soulution was substituted with the fresh one lest the SAS concentration decrease. Procedure of grinding has been detailed in [24].

Particle size distribution functions are generated with laser diffraction microanalyzer. Using X-ray structural analysis, it was demonstrated that particles make up a bcc-iron in nanocrystal state. The grain size defined from lines broadening is (24 ± 4) nm after 1 h of grinding, and (4 ± 1.5) nm after 99 h of grinding. In the course of grinding, the saturation of powder particles with carbon, oxygen and hydrogen atoms occurs [24].

APM-studies performed at the scanning sound microscope P4-SPM-MDT of HT-MDT firm. Silicon cantilevers with the radius of curvature of the needle less than 10 nm and the convergence angle of the needle at its top less than 20° of Silicon Company have been used. The measurements have been performed in air under contact mode measurement of topography and lateral friction forces.

Particle fastening to the backing has been effected in two ways: to study topography features, fastening was in polystyrene; and for correct shape determination and size measurements plasma polymerization technique in the atmosphere of remainder air was used [25].

RESULTS AND DISCUSSION

Shown in Fig. 3 are the size distributions of particles obtained by iron grinding in SAS solution during 24 and 99 h. Their comparison gives that minimal particle size attainable (0.2–0.25 mm) is achieved after 24 h grinding and does not change in the course of subsequent 75 h treatment, which is testimony of availability of mechanisms, restricting the dispersity increase.

Shown in Fig. 4 are images in lateral friction forces of the various particle surfaces after 1 h of grinding, together with lateral forces profiles. Section plane is indicated by line inside the frame. Scanning pitch changed in the range from 0.6 to 1 nm. Steps width in Fig. 4, *a* is equal to about 20 nm, and in Fig. 4, *b* is equal to 100 nm .

Shown in Fig. 5 is 3*D* image of typical particle obtained after 99 h grinding and particle profile. Section plane passes through the top of particle. The particle is disk-shaped with the ratio of height to diameter of 1 : 7 that is in agreement with model concept. Shown in Fig. 5, *c* and *d* are 3*D* image of this particle surface locality and the locality profile. Step width is equal to 50 nm and corresponds to 10 particle sizes, step height is about 5 nm and corresponds to the size of single grain.

Shown in Fig. 6 is 3*D* image and profile of one of the least detected particles. It has the
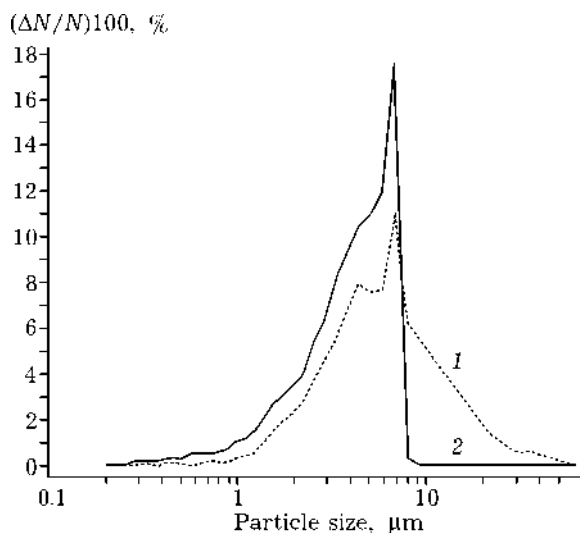
shape of ellipsoid with the axle ratio of 1 : 4 : 8. Minimum diameter of particles, on APM evidence, is 150–200 nm, the height being 70–100 nm. We emphasize that APM method and laser diffractometry (see Fig. 3) give close results as per minimum particle size.

Outlined above, the mechanism of dispersity restriction by effect of superplasticity is not universal. When temperatures are sufficiently low, or when irregularity in nanocrystal structure is reasonably high, or when mobility of atoms on the intergrain boundaries is not-too-high, prerequisites to effect of superplasticity do not necessarily occur. In this case the powder particle will deform by means of macrodislocations transfer, however, the latter ones may then be impeded by various obstacles in the bulk of particle or on its outer boundary. Therewith prerequisites to further particle destruction may occur.

The broad outlines of the grinding process can be then visualized in the following way. When powder particles are reasonably large in size, macrodislocations are not necessarily free to rise to the particle surface: various irregularities in structure or the barrier at the outer surface may impede them. In the process, the buildups of "locked" macrodislocations will be created, on which the heavy strains are concentrated capable of producing structural disruption [26]. As a result, the particle will be ground until its size is less than macrodislocations buildup capable of making disruption. From this condition the minimum powder sizes produced by MA method can be estimated.

A minimum in size buildup of macrodislocations may consist merely of two ones, thus the minimum particle size cannot be less than this buildup size. From the theory of dislocation buildups is well known that their length l is one-to-one correlated with the quantity of dislocations and locking strain t. For the buildup of two dislocations

$$l = \frac{bG}{2\pi(1-\nu)\tau} \qquad (12)$$

The value of minimum length may be obtained setting $t = s_B$ – the strain of disruption
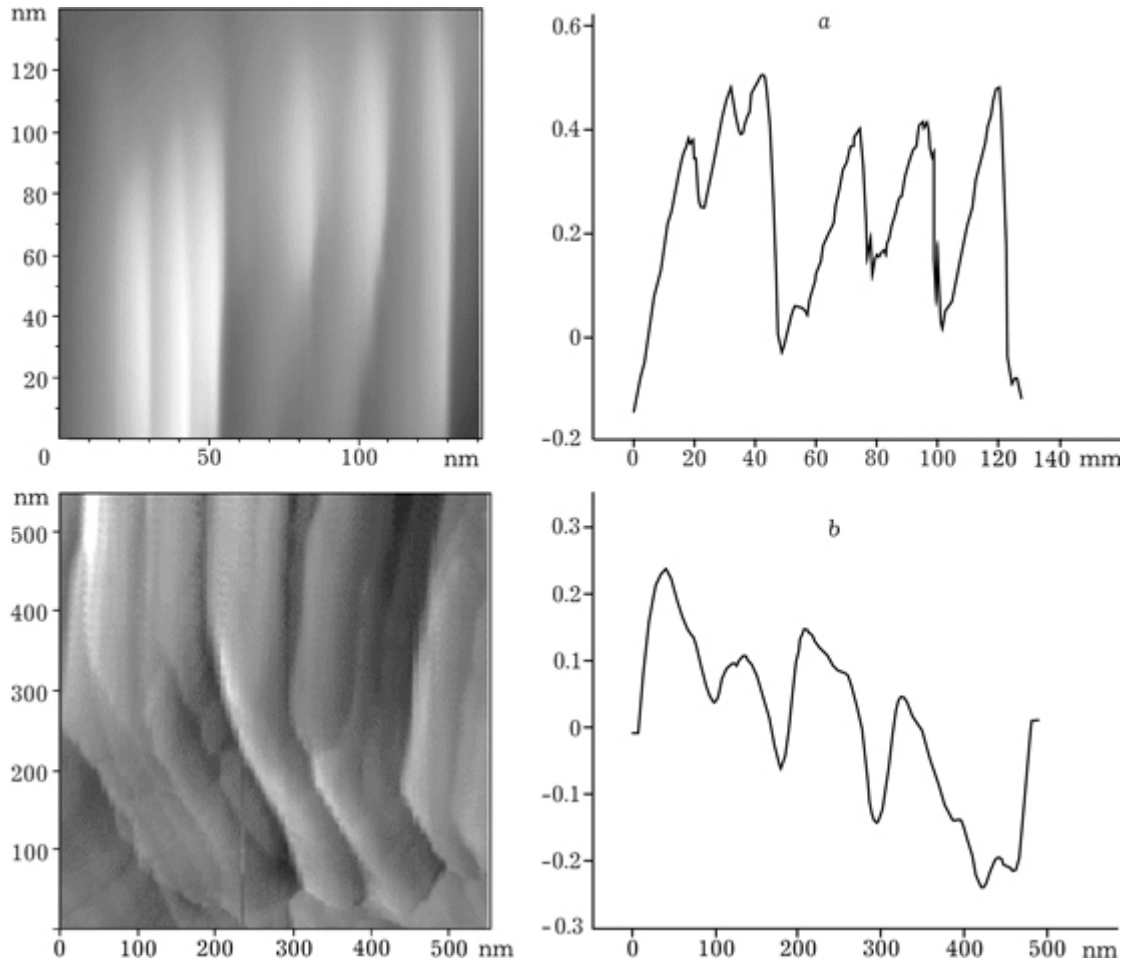
$$l_{\min} = \frac{bG}{2\pi(1-\nu)\sigma_B} \qquad (13)$$

$(\Delta N/N)100$, %

Fig. 3. Size distributions of particles obtained by iron grinding in SAS solution for 24 (*1*) and 99 h (*2*).

Fig. 4. APM images in lateral friction forces and profiles of surface localities of the particles obtained through 1 h grinding: a − steps of one grain width, b − of 5 grain widths.

The direct proof of this relationship for the macrodislocations buildup is difficult to perform and yet, with an eye to analogy between theories of conventional dislocations and macrodislocations, the relationship (12) can be taken as an evaluative one for macrodislocations buildups as well.

The ratio G/s_B is generally falls into the interval $(10-10^3)$ [26]. For average value from this interval $G/s_B \gg 10^2$ and $v \gg 0.5$ formula (12) gives $l_{min} \gg 30b$.

Hence, the minimum size of powder particle available form MA method cannot be less than the size of cube, $30b$ on a side, and its minimum volume is

$$V_{min} = l_{min}^3 = 30^3 V_{HK} \qquad (14)$$

where V_{HK} is the volume of one nanograin of diameter $b = 2R$. The size of grain in particles

produced after 99 h of grinding, on X-ray structural analysis data, is equal to 4 nm, consequently, the minimum particle size must be about 120 nm. The minimum size of particles for just the same powders found by the APM method is equal to 150−200 nm, which accords well with theoretical conclusions. Formula (13) is usable as well to estimate the minimum thickness h_{min} of disk-shaped particles produced under super-plasticity regime. It is well known fact indeed that the slipping lanes while upset are directed at an angle p/4 to surface. Hence we find

$$h_{min} \approx l_{min} \cos \frac{\pi}{4} \approx 20b \qquad (15)$$

The value calculated is also in good agreement with experimental data. It has been found by the APM method that after 99 h grinding, when grain size reaches the value of 4 nm,
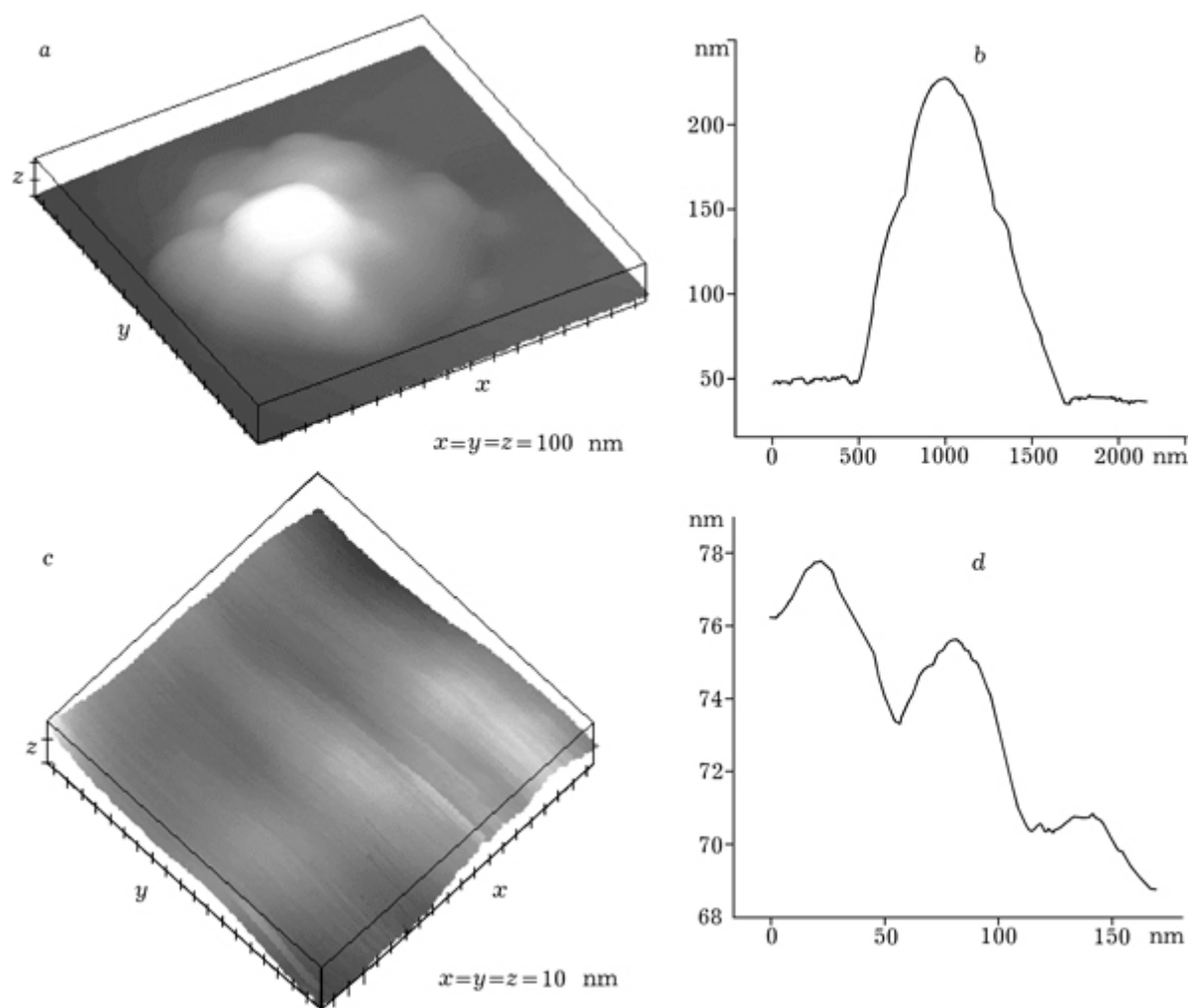
Fig. 5. 3*D* image of particle (*a*) and profile of particle (*b*) obtained after 99 h grinding; 3*D* image of particle surface locality (*c*) and profile of that locality (*d*).
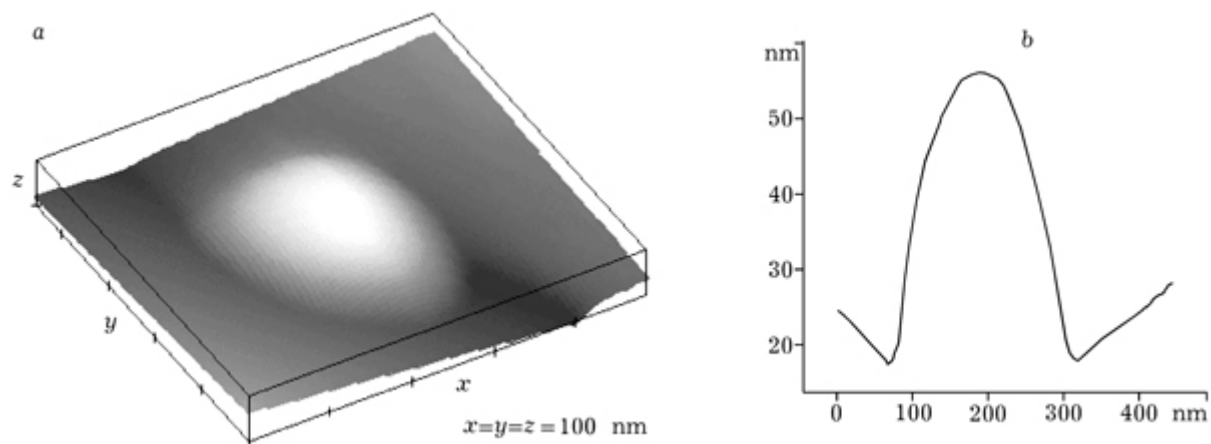
Fig. 6 3*D* image of one among the finest available particles obtained through 99 h grinding (*a*) and profile of the particle (*b*).

the minimum particle height is about 80 nm. Thus one can affirm that the process of grinding and nanostructure formation for powders produced by means of ball mills, as a whole, falls into pattern shown in Fig. 1, *a−e*. In the accompanied experiment, the structure shown in Fig. 1, *e* arises after 1 h grinding. Further action on powder results not only in its mechanical grinding, but also in intensive saturation with admixtures, which are the products of organic media disintegration − carbon, oxygen, and hydrogen. Obviously, saturation occurs along the boundaries of nanostructure and depends on the degree of its development, however, specific mechanisms yet remain to be completely explained and invite a special investigation. Nonetheless, the effect of saturation with admixtures has been observed by a number of other authors [2, 27−30]. It should be mentioned that supersaturation is relatively quick to occur [14, 24, 27−31] to the point where release of particles of new phases containing admixture atoms in the constitution of them is observed (see Fig. 1, *g*). Further treatment results in growth of their volume fraction. As a result, in the course of further grinding, the particle structure comprises the nanostructural multiphase mixture (see Fig. 1, *h*).

CONCLUSIONS

1. A sketch of formation and development of nanostructure and phase constitution of powder particles produced through mechanoactivation in ball mills with SAS has been suggested, which takes account of the process features connected with multiple high-velocity cold-hardening, high temperature polygonization and recrystallization, and multiple re-chill in liquid media.

2. The main structure elements responsible for plastic deformation transfer in nanograined polycrystals are macrodislocations.

3. Under high activity of SAS and well-promoted grain-boundary diffusion the powder particles may transform into super-plastic state. Their further disintegration stops, and particles take a disk shape with minimal thickness $h_{min} \gg 20b$.

4. Under low activity of SAS or/and not-too-high diffusion mobility of atoms at the grain boundaries the phenomenon of superplasticity does not necessarily occur. In this case, plasticity is effected by transfer of macro-dislocations as well, however, they are then subject to stopping at the barriers associated with the structure irregularities, thus forming macro-dislocations buildups terminating in disruption. Under these circumstances a minimum volume of particle is $V_{min} = l_{min}^3$, $l_{min} \approx 30b$.

Acknowledgements

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