

Fine Grinding Intensification Mechanism for Metal Powder Materials

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Abstract—The fine grinding of metal powders is shown to proceed simultaneously with particle aggregation. Here, destruction and aggregation are in dynamic equilibrium. To destroy metal particles during repeated deforming actions, it is necessary to prevent the relaxation of microcracks on the surface of these particles in the intervals between mechanical actions. Therefore, the fine grinding of metal particles without the substances that prevent microcrack relaxation is practically impossible. It is shown that the following two reagents, the mechanisms of influencing the process of which are different, should be used to intensify the fine grinding of metals: a metal defect formation aid at the initial stages of action on the particles to be ground and a dispersant in order to disaggregate the products of disintegration of these particles.

Keywords: metal powders, grinding, microcracks, grinding aids, surfactants

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INTRODUCTION

In recent years, the attention to the methods and processes used for producing fine and ultrafine powders for additive technologies, powder metallurgy, and mineral pigments has been quickened. Obviously, the production of a fine-grained material requires long-term grinding and, consequently, high specific energy, since it is associated with the formation of a very large new grain surface and the inevitable aggregation of forming fine particles. One of the methods to reduce the energy consumption of fine powder production is to use certain grinding aids (GAs) in various technological media.

Currently, GAs are mainly used in the cement industry and the production of pigments. Accordingly, most studies of the mechanism of their action relate to these industries. The reviews of these studies [1–6] demonstrate that the mechanism of action of GAs is still unknown. The studies of the effect of metal powder GAs are scarce, since the scale of use of mechanical metal grinding is not so large and the complexity of experiments is very significant.

EXPERIMENTAL

Our studies and the experiments described in the cited works were carried out using laboratory ball drum mills and vibrational grinding with steel milling balls and using various liquid and gas process media and common surfactants. In experiments, sieve and

optical (laser) methods and optical and electron microscopy were used to determine the powder grain sizes.

RESULTS AND DISCUSSION

The modern concepts of fine grinding of metals and metal alloys down to 100 μm or smaller in milling-body machines can be illustrated in Fig. 1 [7]. Fine grinding proceeds simultaneously with the process of particle aggregation, which is caused by a change in the relation between the effect of the gravitational forces of external fields (gravitational, centrifugal) and surface forces in favor of the latter. Curve 1 in Fig. 1 reflects a decrease in the particle size with increasing milling time, and curve 2 characterizes the aggregation

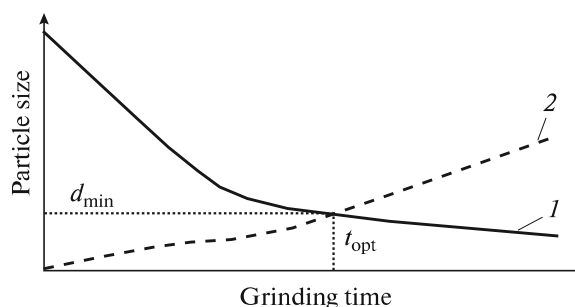


Fig. 1. Schematic diagram of the fine grinding of iron [7].

Table 1. Geometric parameters of medium components and GAs (sizes, nm)

Media		GAs	
gas	liquid	strength reducer	dispersant
AR—0.38	Water—0.30	H ₂ —0.22	Stearic acid— 0.5×0.25
N ₂ —0.37	Isopropanol—0.39	H—0.11	Oleic acid— 0.7×2
O ₂ —0.35	Hexane—0.49	He—0.21	
NH ₃ —0.35		C—0.14	
		N—0.13	
		O—0.12	

process. The intersection point of curves 1 and 2 indicates the minimum achievable particle size (d_{\min}) and the technologically optimal milling time (t_{opt}), at which destruction processes are in dynamic equilibrium with aggregation processes and exceeding of which does not lead to a decrease in the grain size in a milled material. Note that particle aggregation proceeds partly by a mechanism close to cold working, in addition to the main mechanism of formation of a “coat” of thin particles on larger ones (coating), which occurs in the technologies of disintegration of mineral raw materials.

The existence of a limit to the detectable metal particle size during grinding is an experimentally established fact. The minimum particle size during grinding depends not only on deformation conditions, but also on the properties of a material. Experiments showed that each metal has its own minimum particle size (practically achievable for a certain grinding method). For example, under the same grinding conditions, this size is 25 μm for aluminum, 10 μm for nickel, and 5 μm for palladium [8, 9].

The process of fine grinding of solid particles in milling-body apparatus can be divided into the following stages: I, the appearance of lattice defects (dislocations) during shear deformation; II, development of a network of microcracks on the particle surface due to dislocation coalescence and growth; III, microcrack growth to the sizes at which healing (relaxation) is impossible, which causes the destruction of particles; IV, disaggregation of the elements having formed during particle destruction; and V, prevention of aggregation of particle destruction products (note that this is a method of aggregation control rather than a classical stage of grinding).

The phenomenon of plasticity is characterized by the fact that the destruction of an atomic lattice requires an element displacement of 20–50% during shear deformation of ductile metals, unlike minerals (low-plasticity objects), the displacement of atoms in the crystal lattice of which by 1–10% relative to each other causes its destruction. Therefore, the destruction of metal particles, unlike mineral particles, requires repeated exposure to a deforming force,

which determines a significantly longer time for fine grinding of metal particles compared to mineral particles. Thus, fatigue failure of particles occurs when plastic materials are ground. There are three main stages of fatigue failure [10–12]: the formation of nucleation defects, which ends in the appearance of microcracks 0.3–0.6 nm long; the subcritical growth of fatigue microcracks to a size of 1–3 nm; and the accumulation of critical volume of microcracks up to 100–300 nm in size, which creates a stress exceeding the ultimate strength of the metal.

The need to use multiple deforming actions to destroy metal particles requires preventing the healing (relaxation) of microcracks on the surface of these particles in the intervals between mechanical actions. Therefore, fine grinding of metal particles without the use of substances preventing the relaxation of microcracks is practically impossible. These substances provide an adsorption decrease in the strength of metals. It occurs in the following two stages: reagent adsorption on the outer surface of the material to be deformed and on the surfaces of the formed defects; the penetration of the active components of the reagents into the material, i.e., the diffusion of the active components of the medium and reagents to nucleating microcracks, which causes an increase in the volume of the defects formed inside particles.

These processes lead to the adsorption fatigue effect: a change in the fatigue failure resistance of a metal under the influence of adsorbing surfactants on external and developed internal surface defects of the metal under cyclic stresses.

Since the sizes of the nucleation defects that appear in metal particles during shear deformation are only 0.3–0.6 nm due to the steric factor, a very limited number of available substances can be used to wedge these defects by preventing their relaxation. In practice, hydrogen, helium, oxygen, nitrogen, and carbon atoms with geometric dimensions of 0.11–0.22 nm have such properties (Table 1).

The development of a network of fatigue microcracks due to dislocation coalescence and growth leads to an increase in the surface defects to 2–5 sizes of metal lattice parameters, up to 1–3 nm, which allows

Table 2. Effect of grinding medium and aids on the fine grinding of metals

Disintegration stages	Effect of medium	Effect of GAs	
		strength reducer	dispersant
I. Appearance of lattice defects (0.3–0.6 nm)	+	+	–
II. Development of a network of microcracks on the particle surface due to dislocation coalescence and growth (1–3 nm)	+	+	–
III. Microcrack growth to the sizes at which their relaxation is impossible (100–300 nm)	+	+	+
IV. Disaggregation of the elements having formed during particle destruction	+	–	+
V. Prevention of aggregation of particle destruction products.	+	–	+

molecules of gas and liquid medium components 0.35–0.50 nm in size to penetrate into microcracks, promoting an increase in the microcrack volume (Table 2).

The accumulation of critical microcrack volume, which creates a stress exceeding the ultimate strength of the metal, manifests itself in the appearance of surface microcracks 100–300 nm in size and is accompanied by the destruction of metal particles. At this stage, the effect of heteropolar surfactants 1–3 nm in size, which are actively adsorbed at newly formed solid–liquid or solid–gas interfaces, is added to the wedging action of a medium (Table 2).

Dry Grinding

Gorokhovskiy [13] studied the laws of dry grinding of metals in the presence of various organic substances. The grinding of iron particles in a ball mill showed that organic polymers, namely, polyethylene (PE) and polymethylmethacrylate (PMMA), are effective intensifying additives. The author explained this fact by the influence of free radical particles, which form during the mechanothermal destruction of polymer macromolecules, on the deformable surface. Most metals are ductile. Therefore, powder particles undergo dynamic cold working and heat to high temperatures comparable to the melting point during mechanical grinding, when the particles are subjected to a shock compressive load, due to severe plastic deformation. The area of local temperature rise is small, 10^{-3} – 10^{-5} μm^2 , and the lifetime of temperature rises is approximately 10^{-4} s [14, 15]. The pressure at contact points, where shear deformation develops, can reach 1.5–1.8 GPa, and the temperature can exceed 600°C. A local increase in the temperature of the particles leads not only to their deformation, but also to saturation with organic medium decomposition products, namely, oxygen, carbon, nitrogen, and hydrogen.

Interestingly, the addition of PE during grinding in an inert argon atmosphere exerts a greater effect than in air, which the author of [13] explains by the fact that the life of the free radicals having formed during poly-

mer degradation in argon is longer than in the presence of oxygen in air. The maximum efficiency of PE is achieved by adding 5% PE (Fig. 2). At a higher PE content, the grinding efficiency decreases due to the damping effect of the milling medium [13]. A comparison of the efficiencies of various common polymers as GAs for an iron powder showed that the polymers can be arranged in the following efficiency increasing order: polyterephthalate (PTP)–polyethylene (PE)–polyisobutylene (PIB)–polyvinylchloride (PVC)–polystyrene (PS)–polymethylmethacrylate (PMMA).

This row corresponds to the order in which the activation energy of thermal degradation of these polymers decreases: at the same stresses, more intense degradation occurs in polymers with a lower degradation activation energy. These polymers generate a larger number of free radicals at the same action, which affects the process of iron grinding (Fig. 3). The higher efficiency of these polymers as metal GAs can be explained by the fact that the activation energy of their thermal degradation is lower than the dissociation energy of the diatomic gases used as a medium (Table 3).

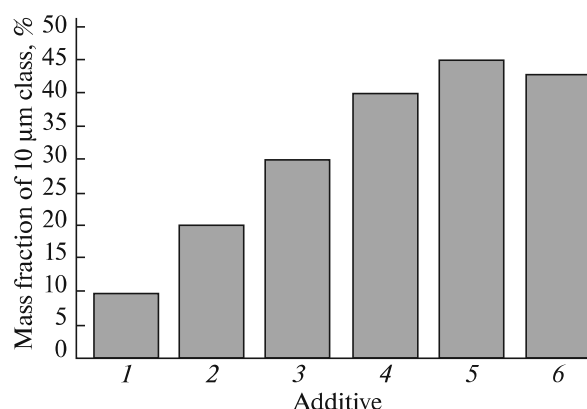


Fig. 2. Influence of additives on the dry grinding of an iron powder [13]: (1) without additive, (2) petrolatum, (3) oleic acid, (4) PE in air, (5) PE in argon, and (6) PMMA in air.

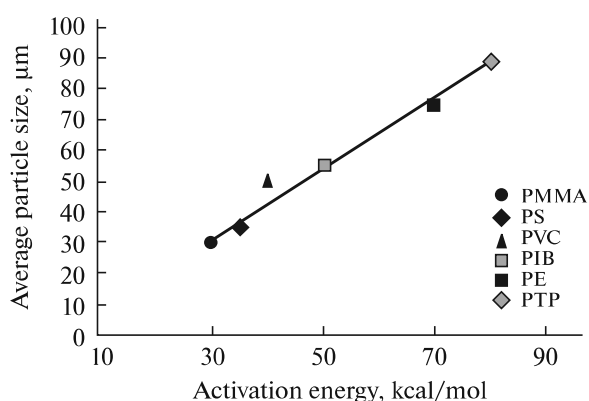


Fig. 3. Refinement of an iron powder vs. the thermal destruction activation energy of a polymer [13].

The powders predominantly containing particles less than 100 μm in size are spatial structures, which determine their structural–rheological properties and their response to mechanical influences. The main structural–rheological characteristic of such systems is fluidity. Therefore, the control of the fluidity of powder is the most important factor in heterogeneous chemical–technological processes, which include powder production by mechanical grinding.

The main feature of structure formation in dispersed systems is the coalescence of particles into aggregates. Since powder particles move in a gas phase, the viscosity of which is low, the resistance to their motion is mainly caused by the contact interaction of the particles, which determines the effect of dry friction.

To break a structured dispersion, it is necessary to reduce the interparticle interaction forces, i.e., to weaken dry friction.

The most important forces determining the interaction of powder particles are capillary, electrostatic, and van der Waals forces.

For highly concentrated dispersed systems (which include finely ground powders), the general physico-chemical principle of controlling their structural–rheological properties is a combination of weakening the adhesion strength of particles in contacts by modifying the surface of dispersed-phase particles mainly with surfactant adsorption layers and by mechanical actions.

As was shown in [1], the adsorption of ethylene glycol vapors on cement particles significantly decreases the adhesive forces between them. The adsorption of aliphatic alcohols and carboxylic acids was found to reduce the specific surface energy of limestone particles, which in turn affects the ability of these particles to aggregate. At the same time, the disaggregation

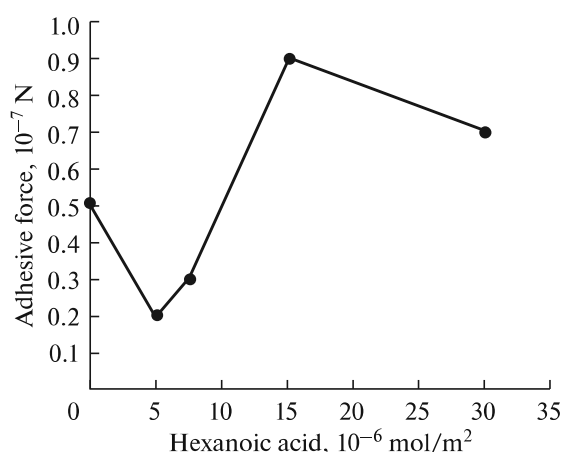


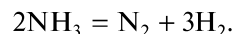
Fig. 4. Effect of hexanoic acid treatment of limestone particles on the adhesive force of individual particles [1].

effect manifests itself in a narrow concentration range of these surfactants: an excess of surfactants enhances particle aggregation (Fig. 4).

The study of the influence of the hydrocarbon radical length of heteropolar surfactants (carboxylic acids) on the dry crushing of limestone and an SmCo₅ alloy showed that surfactants with a radical of 5–8 carbon atoms have the highest activity [1, 4].

This mechanism of interaction of a medium with a solid body during disintegration can easily be applied to the use of hydrogen and nitrogen, which are capable of dissociative adsorption–chemisorption, during which an adsorbed molecule breaks up into two or more fragments. Breaking up into two fragments, diatomic gases bind the interatomic dangling bonds having formed when a microcrack appears, creating surface mechanical compounds, namely, metal hydrides and nitrides.

It was of interest to find GAs capable of acting according to a similar mechanism, but cheaper and easier to use than PMMA. It was logical to assume that the simplest source of nitrogen and hydrogen could be ammonia, which decomposes at a temperature of 270°C,



In addition, ammonia is a strong reducing agent, which would prevent the oxidation of the newly formed surface of metal powders.

As was found in [17, 18], gaseous ammonia interacts with aluminum and titanium at 750–900°C. Moreover, nitrogen and hydrogen formed during the decomposition of ammonia exhibit higher reactivity than the primary gases [17]. This is caused by the fact that the nitrogen molecule has a very strong triple interatomic bond (N≡N) with an energy of 942 kJ/mol, the hydrogen molecule has a binding energy of 432 kJ/mol (H–H), and the ammonia molecule has a binding energy of only 356 kJ/mol (N–H).

Table 3. Dissociation energy of diatomic gas

Gas	H ₂	O ₂	NH	N ₂
Energy, kJ/mol	432.1	493.6	355.9	942.4

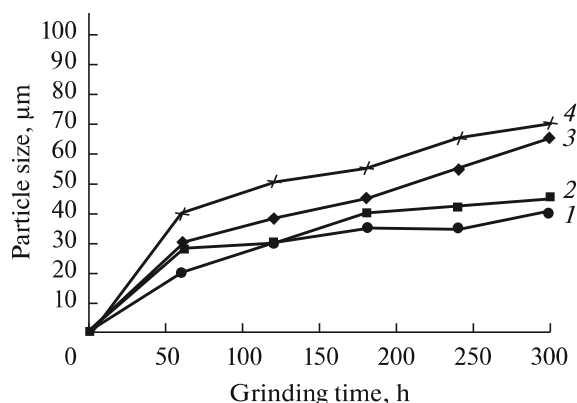


Fig. 5. Effect of (1) nitrogen, (3) ammonia and (2, 4) their mixtures with stearic acid, respectively, on the vibration grinding kinetics of aluminum.

The effect of ammonium chloride on the grinding of an aluminum powder was studied in [21]. Cold welding of aluminum particles was found to occur in the absence of an NH_4Cl additivity, and the introduction of ammonium chloride leads to a significant increase in the specific surface area of the powder and its passivation with respect to oxygen.

The investigation of grinding of aluminum shavings on an IVS-4 vibrating grinding mill using the technique described in [19] showed that the rate of aluminum powder grinding in an ammonia atmosphere is 30% higher than the grinding rate in a nitrogen atmosphere (Fig. 5). The addition of 0.5% stearic acid in the nitrogen atmosphere increases the grinding rate of aluminum by 50%, and that in the ammonia atmosphere, by 90%.

Thus, a combination of metal defect formation aids at the initial stages of action of ammonia on the particles to be disintegrated and a dispersant for the disaggregation of the products of disintegration of these particles (stearic acid) makes it possible to significantly intensify the fine dry grinding of metal powders.

Wet Grinding

The works by Reh binder and his followers [20] showed that the deformation of a solid body in a liq-

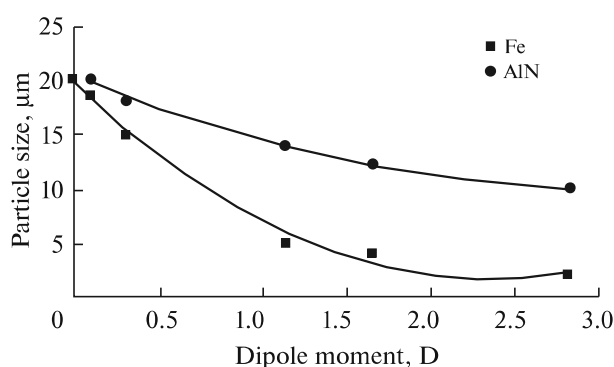


Fig. 6. Effect of the dipole moment of a liquid medium on the fine grinding of AlN and Fe powders [20, 21].

uid, which can wet its surface, causes a significant decrease in the mechanical strength, facilitating destruction (Reh binder effect: an adsorption-induced decrease in strength). The effect of a medium on microcrack nucleation is caused by a decrease in surface energy as a result of the sorption interaction of the surface with the medium. To reveal a relationship between the properties of the liquid phase and the influence on the grinding of metal powders, we can use the data on grinding iron in water, acetone, isopropyl alcohol, acetic acid, oleic acid, and heptane [20–23] and the data on grinding aluminum nitride (AlN) in water, acetone, isopropanol, and hexane [24].

Figure 6 shows the dependence of the results of grinding iron and aluminum nitride particles on the polarity of the liquid medium, which is characterized by the dipole moment of liquid molecules measured in debye (D). These data indicate that the efficiency of grinding hydrophilic metal powders increases with the polarity of the liquid medium (D): hexane, 0; heptane, 0; toluene, 0.3; acetic acid, 1.7; isopropyl alcohol, 1.7; water, 1.9; and acetone, 2.9 (Table 4).

The dipole moment of liquid affects the surface energy of the solid–liquid interface. Its decrease intensifies the grinding process by intensifying the formation and expansion of surface microcracks and the dispersion of newly formed particles. As was shown in [25, 26], the higher the dipole moment of a liquid

Table 4. Characteristics of the liquids used as a metal grinding medium

Liquid	Dielectric constant	Dipole moment, D	Surface tension, 10^{-3} N/m
Water	80.0	1.9	72.8
Acetone	20.7	2.9	23.7
Isopropyl alcohol	18.3	1.7	21.7
Ethanol	24.3	1.7	22.8
Methanol	33.1	1.7	22.6
Hexane	2.0	0.0	18.4
Toluene	2.2	0.3	28.5
Ethylene glycol	37.0	2.2	46.1

Table 5. Effect of medium on the vibration grinding of aluminum shavings for 4 h

Medium	Nitrogen	Isopropyl alcohol	Decane	Decane + stearic acid (0.5%)
Yield of 0.125 μm class	35	78	45	63

medium (i.e., its polarity), the stronger the disintegration of hydrophilic solid materials.

Interestingly, the surface tension of these liquids at the gas–liquid interface has no obvious relation to their effect on the results of metal grinding (Table 4).

The adsorption of polar molecules of a liquid medium decreases the surface energy of solid particles, which provides an adsorption-induced decrease in strength and also prevents the aggregation of fragmentation products, hindering coagulation contacts between them.

Table 5 presents data on the crushing of aluminum shavings in various media: grinding in polar isopropyl alcohol proceeds most efficiently, the grinding parameters in nonpolar decane are close to the data on grinding in a nitrogen atmosphere, and the addition of stearic acid to decane exerts an intensifying effect on grinding.

Interesting data on the ultrafine grinding of iron powders were obtained in [21–23]. Long-term grinding in an inert liquid (heptane) was found to form particle aggregates, as in the case of dry grinding without additives.

During long-term mechanical activation of iron (more than 40 h), the destruction of the organic medium in heptane is observed, which is accompanied by the diffusion of carbon and hydrogen into the surface layers of iron particles. Interestingly, a surfactant (oleic acid) affects the grinding of iron in a nonpolar medium, heptane. The addition of oleic acid to heptane during iron grinding exhibits the maximum effect at a concentration of 0.2–0.5%; an increase in the oleic acid content leads to a decrease in grinding characteristics [27].

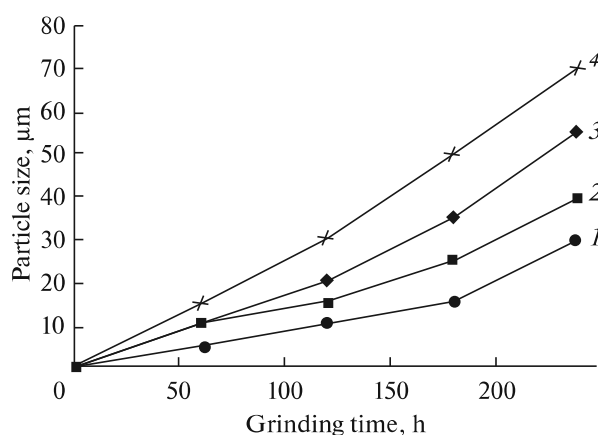


Fig. 7. Effect of urea and stearic acid (SA) additives on the vibration grinding kinetics of steel shavings in isopropyl alcohol: (1) without additives, (2) alcohol + urea, (3) alcohol + urea + SA, and (4) alcohol + urea + SA.

The influence of polymer additives (polyethylene, polystyrene) on the grinding of iron powder in nonpolar media was studied in [28, 29]. Unfortunately, no data on the influence of these additives on the grain size in crushed powders were presented. Nevertheless, it was noted that these polymers actively interacted with the surface of iron particles to form iron carbides, which indicates their fixation in surface microcracks and an inevitable influence on the process of particle destruction under mechanical actions. Moreover, polystyrene exerts a greater effect than polyethylene, which coincides with their effect during dry powder grinding (Fig. 3). The introduction of a surfactant (stearic acid) into this system causes a decrease in the amount of carbon on the particle surface, which is caused by the dispersing effect of the surfactant. This is confirmed by an increase in the hydrophobicity of iron particles, which facilitates their dispersion in a nonpolar medium.

Thus, we can conclude that the character of influence of nonpolar liquid media on the fine grinding of metals is similar to the behavior of nonpolar gaseous media.

The above data on the influence of ammonia on the dry grinding of metal powders suggests that a substance that can be a source of active hydrogen and nitrogen atoms can also be chosen for wet grinding. Such a source of active nitrogen and hydrogen can be urea $\text{CO}(\text{NH}_2)_2$.

The authors of [30, 31] described the production of a titanium nitride powder by joint grinding of titanium and urea in a ball mill. During grinding, urea was found to decompose into isocyanic acid HNCO and ammonia NH_3 . Since the $\text{N}=\text{C}$ bonds in HNCO and the $\text{N}-\text{H}$ bonds in NH_3 are very weak, they break down upon grinding to form active nitrogen and hydrogen, which react with the surface of a metal powder.

A patent search revealed a US patent describing the use of urea to remove oxygen from metal powders [32]. The authors of patent [33] proposed to use urea to intensify the grinding of cement clinker, and the use of a combination of glycol and urea for the same purpose was suggested in patent [34]. The use of urea to intensify the grinding of cement was also proposed in USSR inventor's certificate [35]. However, we failed to find any data on the use of urea to intensify the grinding of metal powders.

The investigation of the grinding of steel shavings on an IVS-4 vibrating grinding mill in isopropyl alcohol using the technique described in [19] showed that the addition of urea (2%) increases the grinding rate by 40%, and the use of a combination of urea and stearic acid makes it possible to increase the grinding rate by almost 3 times (Fig. 7). These results support the effi-

ciency of using a combination of the following GAs for defect formation at the initial stages of action on the particles to be fragmented: urea and a dispersant for the disintegration of the products of particle fragmentation and stearic acid for the intensification of the wet fine grinding of metal powders.

CONCLUSIONS

(1) During fine grinding, the plasticity of metals requires 2–5 times more crystal lattice deformation for the destruction of particles than for brittle materials. Therefore, the grinding of metal particles requires repeated deforming actions, which takes much longer grinding time than for brittle materials.

In order to accelerate the accumulation of defects sufficient to achieve the ultimate strength of metal particles, i.e., to achieve fatigue failure, it is necessary to prevent the relaxation of microcracks having formed during deforming action. For this purpose, it is advisable to use additives of the substances that can provide an adsorption-induced decrease in the strength of metal.

(2) Since the sizes of the nucleation defects that appear in metal particles during shear deformation are only 0.3–0.6 nm due to the steric factor, a very limited number of available substances can be used to wedge these defects by preventing their relaxation. In practice, hydrogen, helium, oxygen, nitrogen, and carbon atoms with geometric dimensions of 0.11–0.22 nm have such properties.

The sources of wedging components on grinding metal particles can be gases, namely, hydrogen, helium, oxygen, carbon dioxide, and ammonia, and solids (or their solutions), namely, urea, ammonium salts, polymethylmethacrylate, polystyrene, and polyethylene.

(3) The action of defect formation aids in metal particles manifests itself in their penetration into a material, which promotes the development of a network of microcracks and leads to the inevitable contamination of grinding products with metal hydrides, nitrides, oxides, and carbides.

(4) The development of a network of fatigue microcracks due to dislocation coalescence and growth leads to an increase in the surface defects to 2–5 sizes of metal lattice parameters (1–3 nm), which allows molecules of gas and liquid medium components 0.35–0.50 nm in size to penetrate into microcracks, promoting an increase in the microcrack volume.

The accumulation of critical microcrack volume, which creates a stress exceeding the ultimate strength of the metal, manifests itself in the appearance of surface microcracks 100–300 nm in size and is accompanied by the destruction of metal particles. At this stage, the effect of heteropolar surfactants 1–3 nm in size, which are actively adsorbed at newly formed solid–liquid or solid–gas interfaces, is added to the wedging action of a medium.

(5) The fixation of heteropolar surfactants on the newly formed surfaces of metal particles promotes the disaggregation of destruction products and prevents their possible aggregation in the turbulent medium of milling devices.

(6) Based on the aforesaid, we can conclude that the following two reagents, the mechanisms of influencing the process of which are different, should be used to intensify the fine grinding of metals: a metal defect formation aid at the initial stages of action on the particles to be disintegrated and a dispersant for the disaggregation of the products of disintegration of these particles.

(7) The efficiency of the action of a liquid medium on the fine grinding of both ductile metals and brittle materials depends on its polarity. When hydrophilic heteropolar metals are ground, the efficiency of a liquid medium increases with the dipole moment of its molecules. The character of the action of nonpolar liquid media on the fine grinding of metals is similar to the behavior of nonpolar gaseous media.

ABBREVIATIONS AND NOTATION

Gas	grinding aids
PE	polyethylene
PMMA	polymethylmethacrylate
PTP	polyterephthalate
PIB	polyisobutylene
PVC	polyvinylchloride
PS	polystyrene

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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