Journal of Engineering Physics and Thermophysics, Vol. 65, No. 4, 1993

INFLUENCE OF GRINDING OF A TITANIUM POWDER ON EXPLOSION CHARACTERISTICS OF ITS AEROSUSPENSION

E. Ya. Shafirovich and O.V. Roslyakova

UDC 541.12.03

The influence of grinding of titanium powders in a planetary mill on the explosion characteristics of their aerosuspensions is studied experimentally. The change in the size, shape, and degree of oxidation of the particles when ground is monitored by a laser size analyzer, electronic microscopy, and neutron activation analysis, respectively. These factors cause the observed intricate dependences of explosion characteristics on grinding time.

Introduction. Mechanical action on solid reagents is used as a means to speed up chemical reactions of the solid-solid, solid-liquid, and solid-gas type. As a rule, in mechanicochemistry this action is performed in the synthesis process. However, preliminary grinding may also affect the rate of chemical reactions [1]. It is known, for example, that lengthy (up to 120 h) grinding of a titanium powder in a ball mill increases its burning rate in nitrogen and decreases the critical ignition temperature in air [2]. The authors have connected this effect with the growth in the number of defects in the surface layer of the particles, which has been supported by the increase in the intensity of exoemission of electrons.

The present article deals with the influence of grinding of a titanium powder in a planetary mill on the explosion parameters of its aerosuspension. Explosion of a sprayed metal powder cloud can be used to produce ultradispersed ceramic particles [3]. The chemical metal-gas reaction proceeding both on the surface of the metal particles and in the gas phase is a heat release source during explosion of a gas suspension. The heterogeneous reaction is governing, at least at the initial stage of the process, since ignition energy is less than that required for a evaporation of a noticeable amount of metal. Preliminary grinding of a metal powder can alter the heterogeneous reaction rate and thus influence the explosion parameters.

The influence of grinding of a powder on the explosion characteristics of its aerosuspension is also of interest from the standpoint of explosion hazard of SHS technologies, where powders are, as a rule, subjected to mechanical action during preliminary operations.

Experimental Procedure. We studied: 1) titanium powder prepared by calcium hydride reduction (hereinafter designated as C); 2) titanium powder prepared by magnesium thermal reduction (hereinafter designated as M). Grinding was performed in an AGO-2 planetary mill with power 1.1 kW at a drum rotation frequency of 2220 rpm and a carrier rotation frequency of 1090 rpm with stainless steel 10-mm-diameter spheres in an air medium. The drum containing the ground powder was opened after it had been cooled to room temperature. The grinding time was varied from 1 to 40 min.

The particle size distribution before and after grinding was determined by a Fritsch laser analyzer. The particle shape was examined under a JEOL Superprobe microscope. Neutron activation analysis was used to determine the oxygen concentration in the powders. The explosion characteristics, namely, lower concentration limit of explosibility (LCLE), maximum explosion pressure, and maximum rate of the pressure increase, were determined on a standard setup of VNIIPO, comprising a 4-liter cylindrical chamber having a conic sprayer and incandescent spiral ignition.

Results and Discussion. Figure 1a shows the LCLE as a function of grinding time. It is seen that grinding for 2-3 min sharply decreases the LCLE of both types of powder. With the grinding time doubled, the LCLE is

---

Institute of Structural Macrokinetics of the Russian Academy of Sciences, Chernogolovka. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 65, No. 4, pp. 399-402, October, 1993.

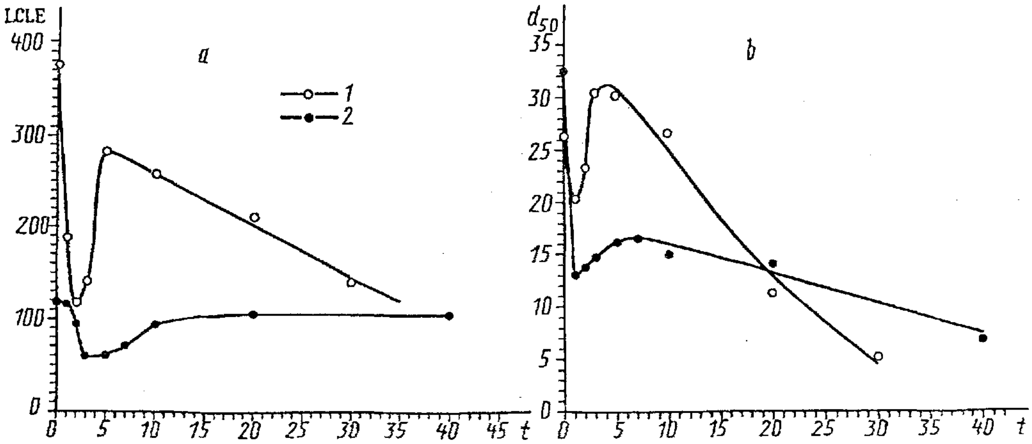

Fig. 1. LCLE, g/m^3 (a), and the median diameter d_{50}, μm (b), of powders C (1) and M (2) as a function of grinding time t, min.

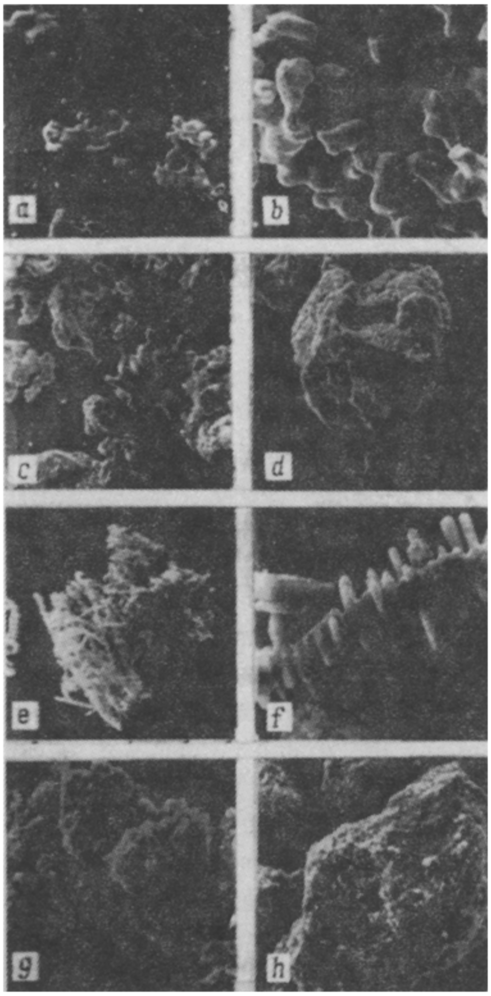

Fig. 2. Microphotos of particles of both powders C (a, b, c, d) and M (e, f, g, h) for different grinding times t and at different magnifications: a) t = 0, ×400; b) t = 0, ×1000; c) t = 1 min, ×400; d) t = 30 min, ×1000; e) t = 0, ×1000; f) t = 0, × 4000; g) t = 2 min, ×2000; h) t = 40 min, ×1000.

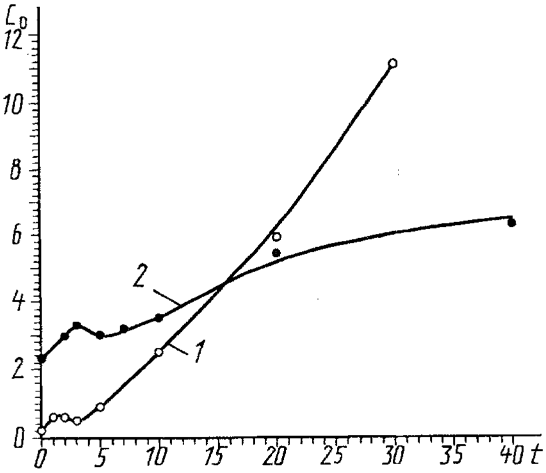

Fig. 3. Oxygen concentration C_O (wt.%) in powders C (1) and M (2) as a function of grinding time t, min.

seen to increase almost to its initial value. A further increase in grinding time gradually decreases the LCLE of powder C and virtually does not influence the LCLE of powder M.

As would be expected, it was found that the maximum explosion pressure does not depend on the grinding time. This is attributed to the fact that the maximum pressure is determined by the total amount of heat released in the system, the thermophysical parameters of the substances, and the conditions for heat exchange with the vessel walls and does not depend on the dynamics of the process.

The rate of pressure increase reflects the dynamics of explosive combustion and obviously must depend on the rate of the chemical reactions proceeding in the system. This is the cause of the strong dependence of the rate of pressure increase on the grinding time t. The largest rate is seen for the time when the minimum LCLE is attained. For example, for powder M the rate of pressure increase at a concentration of 350 g/m^3 increases from 140 atm/sec at t = 0 to 500 atm/sec at t = 3 min.

Analysis of the histograms of the particle size distribution shows that the normal distribution holds true over almost the entire grinding time range. Only with the largest time t = 30 min for powder C, is a noticeable smoothing of the distribution function seen, due to an increasing fraction of fine particles. Figure 1b shows the median diameter d_{50} for both powders as a function of grinding time. It is seen that grinding for 1-3 min already results in an appreciable size decrease. The subsequent growth of the particle size seems paradoxical. For powder C the particle size at t = 5 min even exceeds the initial size. This effect is explained by particle agglomeration and has been noted previously [1]. Lengthy grinding of powders again leads to size decrease. For powder M the agglomeration effect is much less pronounced.

Comparison of the LCLE dependences (Fig. 1a) with the d_{50} ones (Fig. 1b) on the grinding time shows that the size change is probably one of the fundamental factors exerting an influence on the explosion characteristics. However, the correlation of the above dependences is incomplete. For powder M one-minute grinding already sharply decreased the size; however, the LCLE in this case virtually did not change. Also, an increase in the LCLE at t = 3-7 min can hardly be connected with the insignificant size growth due to agglomeration. Conversely, for powder C the LCLE increase at t = 5 min is not as substantial as the size growth. These discrepancies, as well as the strong dependence of the LCLE on the type of powder, point to a substantial influence of other factors, besides the size, on the explosion parameters.

Figure 2 shows microphotos of particles of both powders in the initial state and for different grinding times. First, it is seen that the particle shape of the initial powders is considerably different. For powder C (Fig. 2a, b) the particles look like fused fragments with a highly divided surface, while for powder M (Fig. 2e, f) the particles are shaped like dendrites and their conglomerates. Second, it is seen that grinding causes the particle shape to change greatly (Fig. 2c, g), the largest change being observed for small grinding times. Grinding for 30-40 min

(Fig. 2d, h) results only in breakup of the particles while their shape changes slightly. Thus, the change in the particle shape could be of great importance for decreasing the LCLE for small grinding times.

Since grinding was performed in an air medium, increasing its duration must increase the degree of oxidation of the particles. This is supported by changes in the oxygen concentration in the powders. Figure 3 shows oxygen concentration dependences on the grinding time. Local maxima of these dependences at small times correspond to minima of the LCLE dependences on t (see Fig. 1a) and are, apparently, attributable to an increase in the specific powder surface. As the grinding time increases, the oxygen content grows, attaining 11% for powder C at $t = 30$ min. However, this does not cause the LCLE to grow (Fig. 1a), which points to the relatively weak influence of an oxide film.

Conclusions. Grinding of a titanium powder substantially affects the explosion parameters of its aerosuspension. A sharp decrease of the LCLE and an increase of the rate of pressure increase are seen for short grinding times (2-3 min). This effect should be allowed for when estimating the explosion hazard of production processes where metal powders are subjected to mechanical action.

In conclusion, the authors are grateful to V. I. Rozenband for participation in formulating the problem and to V. V. Kireiko for carrying out the neutron activation analysis of the powders.

REFERENCES

1. E. G. Avvakumov, Mechanical Procedures for Activating Chemical Processes, Novosibirsk (1985).
2. S. I. Voyuev, V. M. Maslov, S. V. Poletaev, et al., Fiz. Goreniya Vzryva, **13**, No. 3, 18-22 (1983).
3. K. Ota and S. Abe, Process for Producing Ultra-fine Ceramic Particles, European Patent O 151 490 A2 (1985).