layer water adsorption occurs, even at a pressure $p_{H_2O} \leq 10^{-5}$ torr, almost instantaneously, in 10^{-5} sec [6], whereas argon diffusion in air, let alone from pores, is a slower process ($D_{Ar-Ar} = 0.2$ cm²/sec) [7].

And so, as a result of rapid adsorption the pores can become sealed by hydroxyl groups (OH^-) or water molecules before all the argon escapes from them. This, of course, can occur only with pores of a certain size, probably not greater than the length of a weak hydrogen bond (4 Å) [8]. If the pores are of a diameter such that isolated hydroxyl groups or isolated water molecules become adsorbed on their surfaces, the argon has time to escape from them.

It should be noted that a powder retains argon for a quite long time (not less than 5 days) after exposure to an argon stream. The diffusion of the argon through the adsorbed water layer is, as would be expected, a relatively slow process. The highest rate of argon removal from an aluminum powder is observed at a temperature of 230-250°C (Fig. 4), which virtually coincides with the temperature corresponding to the maximum rate of water liberation in molecular form.

Thus, the experimental data obtained show that the nature of the annealing environment has a strong influence on the adsorption of the H_2O forming on a powder of the system $Al-Al_2O_3$. The amount of adsorbed water remaining after annealing in relevant environments decreases in the order vacuum → argon.

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SHAPING OF POWDER PARTICLES DURING THE ATOMIZATION OF A MELT WITH WATER

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UDC 621.762.224

Some of the basic laws governing the process of drop shaping during the atomization of a molten metal jet with a compressed gas were formulated in [1-3]. The powder particle shape was found by starting with the ratio between the rates of the processes of spheroidization and cooling of atomized elements of the melt.

The time taken for a particle to cool down to the melting temperature was determined from the expression

$$\tau_1 = d_d \frac{c_m \rho_m}{6\alpha} \ln \frac{t_d - t_g}{t_{me} - t_g}, \tag{1}$$

and the time required for the evolution of the latent heat of solidification, from the expression

$$\tau_2 = \frac{Q}{q \cdot S_d} \tag{2}$$

Institute of Materials Science, Academy of Sciences of the Ukrainian SSR. Translated from Poroshkovaya Metallurgiya, No. 9(165), pp. 5-10, September, 1976. Original article submitted November 27, 1975.

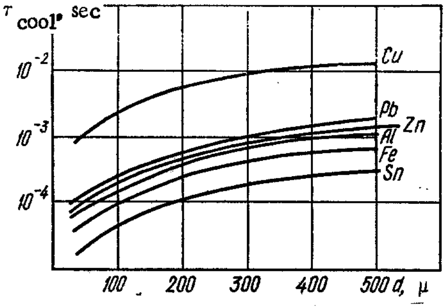

Fig. 1

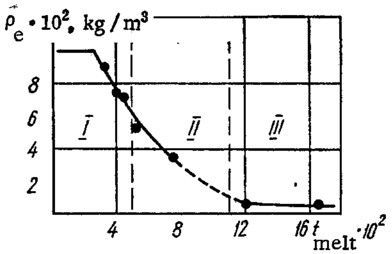

Fig. 2

Fig. 1. Variation of cooling times of molten metal drops with drop size in atomization with water.

Fig. 2. True density of energy carrier in contact with melt being atomized with water under high pressure.

The time taken for the particle to become spheroidized under atomization conditions was calculated with the formula

$$\tau_{sph}=\frac{3\pi^2\mu}{4V_d\sigma}(R_d^4-r_{st}^4). \tag{3}$$

Here d_d, V_d, t_d, R_d, and S_d are, respectively, the diameter, volume, temperature, radius, and surface area of the drop; c_m, ρ_m, σ, μ, and t_{me} are, respectively, the specific heat, density, surface tension, viscosity, and melting point of the metal; Q is the latent heat of solidification; q the thermal flux; α the coefficient of heat transfer from the drop to its environment; and t_g the temperature of the environment.

Comparison of calculated values of τ_1 and τ_{sph} for molten Cu, Pb, and Al showed that in atomization with a compressed gas spheroidization times for these metals are 2.5-3.5 orders of magnitude shorter than cooling times [1], which means that powders of these metals should have a spherical particle shape. In actual fact, however, particles of many powders were found to be nonspherical, often jagged. It was established that the irregular particle shape was due in this case to the formation of refractory oxides on the surfaces of the particles, which prevented their spheroidization.

Calculations of the drop deformation process and data obtained by processing results of experimental studies enabled certain general principles to be formulated underlying the shaping of powder particles during the atomization of a melt with a gas stream.

The conditions of heat transfer from drops during atomization with water differ substantially from those obtaining during atomization with a gas stream, mainly in that heat transfer occurs much more rapidly through water than through a gaseous phase [3, 4]. In an atomization process in which the temperature of the melt is high enough to cause water to boil on the surfaces of its drops three basic mechanisms of heat exchange are possible: 1) when the temperature at the melt–water contacts does not exceed 350°C; 2) when the temperature in the contact zones exceeds 650-700°C; 3) when the melt temperature lies in the range 350-650°C.

In the first of these cases, where the temperature of the contact zones is close to or lower than the critical point of water under a pressure of 50-100 atm gauge, boiling takes place with ebullition, and the coefficient of heat transfer is a maximum [4, 6]. In the second case, where the melt temperature in the contact zones substantially exceeds the "critical" point of water, stable film-type boiling occurs, with a comparatively low coefficient of heat transfer α [4]. The third case represents an intermediate state, in which the temperature conditions prevailing in the contact zones bring about mixed, film-and-bubble boiling. The coefficient of heat transfer under conditions of boiling with pronounced ebullition (bubble formation) can be determined with good accuracy using Kutateladze's empirical formula [6],

$$\alpha_{conv}=\xi p^{0.4}\cdot q_1, \tag{4}$$

where ξ is a coefficient characterizing the type of boiling and the nature of the liquid (in our case $\xi=2.6$), p is the pressure, and q_1 is the thermal load, kcal/m² · h. The quantity q_1, which depends on the physical conditions obtaining in the water–hot-surface contact zones, is given by the expression

$$q_1=10^3 k\beta\sqrt{\sigma_w\rho_s^2(\rho-\rho_s)}\,. \tag{5}$$

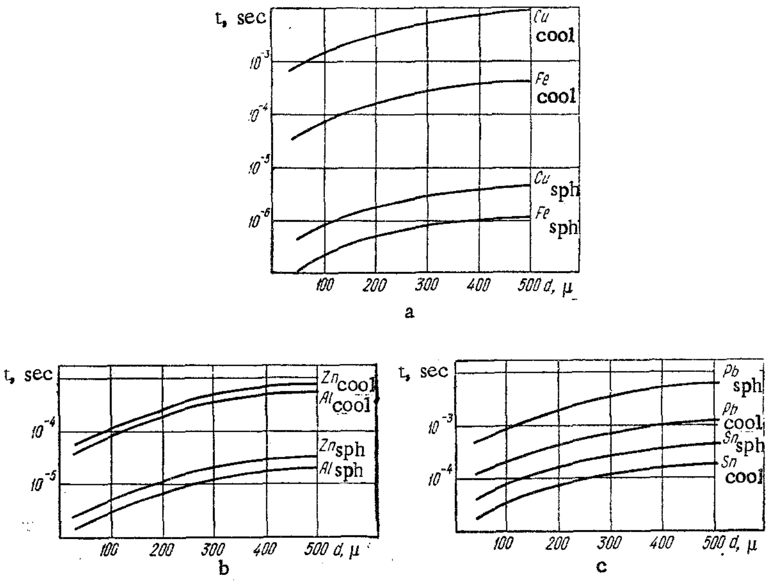

Fig. 3. Comparison of curves of τ_{cool} vs d_s and τ_{sph} vs d_d for various metals in atomization with water: a) $\tau_{\text{cool}}/\tau_{\text{sph}} \gg 1$; b) $\tau_{\text{cool}}/\tau_{\text{sph}} \simeq 1$; c) $\tau_{\text{cool}}/\tau_{\text{sph}} < 1$.

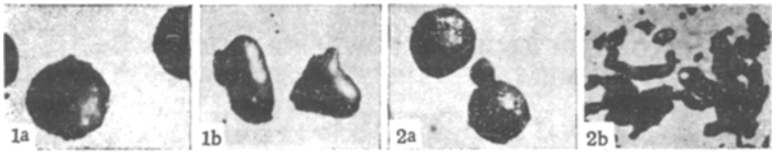

Fig. 4. Powder particles produced by atomizing melts of a) iron and b) tin with 1) compressed air and 2) water under high pressure.

Here k is a coefficient (k=0.16), β is the heat of steam formation (β_W=530 kcal/kg), σ_W is the surface tension of water ($\sigma_W = 77 \cdot 10^{-4}$ kg/m), and ρ_W and ρ_S are, respectively, the densities of water and the steam (ρ_W=1000 and ρ_S=50 kg/m^3). Using the calculated value $q_i = 3 \cdot 10^6$ W/m, we can determine the value of α_{conv} applicable in our case,

$$\alpha_{\text{conv}} = 2.6 \cdot 100^{0.4} (3 \cdot 10^6)^{0.7} \simeq 10^6 \text{ W/m·deg C}.$$

For the case of film-type boiling, according to [6], $\alpha_{\text{conv}_{\text{III}}} = 5 \cdot 10^4$ W/m · deg C, and the intermediate range is characterized by the value $\alpha_{\text{conv}_{\text{II}}} = 10^5$ W/m · deg C.

Now in the heat transfer scheme under consideration very small volumes of molten metal (d_d = 100-500 μ) are involved, and it is therefore necessary to assess the part played by molecular heat exchange, which may prove to be substantial, since the coefficient of heat exchange by conduction, α_{cond}, is inversely proportional to the drop size. This conclusion follows from Nusselt's so-called limiting law [7], which gives the minimum value of the coefficient α_{cond} for a spherical particle,

$$\alpha_{\text{cond}} = \frac{2\lambda_s}{d_d\left(1 - \frac{d_d}{d_s}\right)}, \tag{6}$$

where d_d and d_s are, respectively, the diameters of the drop and its cooling steam shell and λ_s is the thermal conductivity of the steam.

Let us assess the value of α_{cond} in our case [5], where $d_s = 1.15\, d_d$. Calculation shows that, for a thermal conductivity of steam $\lambda_{s(600°\text{C},\ 100\ \text{atm. gauge})} = 8010^{-2}$ W/deg C and a drop size of 100-500 μ, the coefficient of molecular heat transfer is $\alpha_{\text{cond}} = 2 \cdot 10^{-3}$-$10^{-4}$ W/m · deg C, since the value $\alpha_{\text{cond}} = 10^4$ W/m · deg C is a whole order of magnitude smaller than $\alpha_{\text{conv}_{\text{I}}}$ and $\alpha_{\text{conv}_{\text{III}}}$ and is comparable only with $\alpha_{\text{conv}_{\text{II}}}$. Calculated values of coefficients of heat transfer are given in Table 1.

TABLE 1. Calculated Values of Coefficient of Heat Transfer (W/m • deg C) in Atomization of Melts with Water

Temp. in contact zone, °C	Character of boiling (zone No.)	Value	
		formula	magnitude
300—355	I	$\alpha E\text{-}\alpha_{\text{conv}}$	10^6
350—650	II	$\alpha E\text{-}\alpha_{\text{conv}}$	10^5
650	III	$\alpha E\text{-}\alpha_{\text{cond}}+\alpha_{\text{cond}}$	$6 \cdot 10^4$

TABLE 2. Physical Characteristics of Molten Metals and Atomization Spray

Metal	σ, dyn/cm	c, cal/g • deg C	$\mu \cdot 10^3$, kg/m • sec	ρ, kg/m^3	t_{me}, °C	t_{melt}, °C	t_{steam}, °C	α, W/m • deg C
Iron	1500	0,10	0,7	7800	1550	1650	900	$6 \cdot 10^4$
Copper	1200	0,91	3,0	8900	1090	1200	650	$6 \cdot 10^4$
Aluminum	600	0,21	1,2	2700	660	780	450	10^5
Zinc	720	0,09	1,0	7100	420	520	300	10^5
Lead	430	0,04	2,5	11350	325	430	270	10^5
Tin	520	0,05	1,4	7300	230	330	210	10^6

Let us calculate the times taken for 50- to 500-μ-diameter drops of melts with different melting points – Fe, Cu, Al, Zn, Pb, and Sn – to cool down. The physical characteristics of these metallic melts are given in Table 2. Using Eq. (1), we calculate the times taken for such drops to cool down to the melting points, t_{cool}, making the assumption that, under the conditions of cooling of the melts with water, drop deformation ceases as soon as their melting points are reached. The results of this calculation are depicted in Fig. 1.

Next, let us assess the times of drop spheroidization for the melts investigated during their atomization with water. Here it should be noted that a stream of water or a steam emulsion inhibits the process of drop spheroidization much more effectively than does a stream of a compressed gas [3]. The density of the energy carrier will vary depending on the character of boiling and the intensity of steam generation. In accordance with the scheme adopted in our analysis, recourse is had to certain assumptions in order to estimate approximately the true density of the energy carrier in each of the cases under consideration (Table 3).

In Fig. 2 is shown the true density of the energy carrier in contact with the melt being atomized with water, plotted as a function of melt temperature in the atomization zone. While it is not anticipated that calculated and actual values will be in good agreement with each other, the curve nevertheless objectively reflects the phenomena occurring in the metal–steam–water spray. At the same time the graph makes it possible to approximately assess the particle shape of powders produced by atomizing various melts. Thus, nonspherical, jagged particles can be expected to be obtained in zone I and spherical particles in zone III, while zone II is a transition zone in which both spherical and irregular-shaped particles can form, although the appearance of spherical or round particles is the more likely. From this curve it follows that, by exercising control over the contact region between the melt and water (steam) so as to remove completely or partially the steam film enveloping each element of the melt, one can cause the particle shape to deviate from spherical. It should also be possible to determine experimentally the variation of the particle shape coefficient φ with the actual density of the energy carrier in the contact zone, which in turn will be a function of parameters of the energy carrier, i.e., water, and of the geometry of the atomizing device.

To calculate the time taken to spheroidize particles, we use Eq. (3), derived earlier, introducing into it a correction factor allowing for the density of the energy carrier,

$$\tau_{\text{sph}} = \frac{3}{4} \frac{\pi^2 \mu \rho_e}{V_d \sigma \rho_{\text{air}}} (R_d^4 - r_{\text{st}}^4). \qquad (7)$$

It is interesting to compare calculated values of τ_{sph} for the metals under consideration with the values of τ_{cool} for the same elements (Fig. 3). The curves in Fig. 3 can be divided according to the ratio $\tau_{\text{cool}}/\tau_{\text{sph}}$ into three groups:

TABLE 3. Ture Density of Energy Carrier in Atomization of Molten Metals with Water

Melt	Character of boiling	Steam : water ratio in energy carrier	Density of energy carrier ρ_e, kg/m^3
Iron	Film-type	100% of steam	50
Copper	"	The same	50
Aluminum	Intermediate (close to film-type)	70:30	335
Zinc	Intermediate	50:50	525
Lead	Intermediate (close to bubble-type)	30:70	715
Tin	Bubble-type	10:90	905

1. Iron and copper. For these elements τ_{cool} exceeds τ_{sph} by more than one order of magnitude (two orders for Fe and 3.5 orders for Cu).
2. Aluminum and zinc. For these metals the ratio $\tau_{\text{cool}}/\tau_{\text{sph}}$ has the value 10-15.
3. Lead and tin. With these metals the situation is reversed: $\tau_{\text{cool}}/\tau_{\text{sph}} = 0.1$.

Examination of the ratio $\tau_{\text{cool}}/\tau_{\text{sph}}$ for drops of Fe, Cu, Al, Zn, Pb, and Sn melts atomized with water under high pressure shows that complete spheroidization of drops can be expected to occur in the water atomization of such metals as iron and copper. In the atomization of aluminum and zinc, too, powder particles of spherical shape will be produced, but the probability of this happening is much smaller. Thus, any fluctuations in the system, such as oxidation of drops or stripping of steam envelopes by the jet of water, may lead to the appearance of angular or even jagged particles. The atomization of molten low-melting-point metals such as tin and lead will result in the formation of nonspherical powder particles. This is of great importance in the choice of atomization parameters because, as pointed out above, the shape of powder particles can be varied by changing the size of their contacts with steam films. In the case under consideration such a factor is provided by the temperature at which the melt is atomized: The lower the temperature, the greater is the probability that particles of nonspherical shape will be formed.

Special experiments were carried out in which iron and tin melts were atomized under comparable conditions with compressed air (4-5 atm gauge) and water under a pressure of up to 60 atm gauge. These experiments afforded evidence in support of our conclusions, based on theoretical calculations, concerning the particle shape of atomized powders (Fig. 4).

CONCLUSIONS

The particle shape of a powder produced by the atomization of a molten metal with water under high pressure, unlike that of a powder atomized with a compressed gas, is determined by the actual density of the energy carrier (the steam : water ratio) in the contact zone, and can vary within wide limits, from spherical to jagged. The actual density of an energy carrier consisting of water and steam is a function of the temperature of the melt and also of the pressure of the water and the geometry of the atomizing device.

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