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Characterization of Ni-Cu alloy powders produced by the atomization process

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Abstract

The atomization techniques is an extensive way for producing rapidly cooled metal powders. This paper presents an experimental study of some process parameters on the powder properties of a 70 wt.% Ni-30 wt.% Cu alloy. The experimental procedures utilized and the experimental results are presented. Water and air were used as atomizing fluids. The atomizing fluid pressure and the melt pouring temperature were also changed. The following powder characteristics were measured: particle shape and particle size distribution, loss of H_2, apparent density and flowability, and particle microstructure. The powder characteristics are correlated with the process parameters. As was expected, water promotes a greater cooling rate than air in the atomizer and particle shape changes were also noted. An increase in the melt pouring temperature and fluid pressure changes the particle size distribution with a decrease in the mean particle size.

1. Introduction

Atomization is a well-known process in metallurgy by which pure metals and alloys can be obtained in the form of fine powders. Particles produced from liquid metals undergo relatively high cooling rates ranging between 10^2 K s^{-1} and 10^4 K s^{-1} [1].

One of the principal uses of the powders is in powder metallurgy. As a manufacturing method, powder metallurgy either has to compete with other production processes or it may happen to be the only way of fabrication of a certain product [2]. The quality of the powder has a strong influence on both the succeeding manufacturing procedure and the final properties of the end products. It is therefore a major task to control the characteristics of the powders produced by atomization.

The atomization process consists of the formation of liquid metal droplets which undergo rapid solidification. It is achieved by pouring the liquid metal at a conveniently high temperature. The running fillet of metal is disintegrated by a jet of another fluid which pulverizes the liquid metal, as shown schematically in Fig. 1. The metal droplets are so small that they are quenched by the high speed, cool jet of the atomizing fluid and the particles are rapidly solidified. The most common fluids are water, air or an inert gas [3]. The viscosity, density and cooling capacity of water are higher than those of gases, so water as an atomizing fluid produces irregular particle shapes. Gases help to form rather spherical particles. Metals of high reactivity tend to oxidize with both water

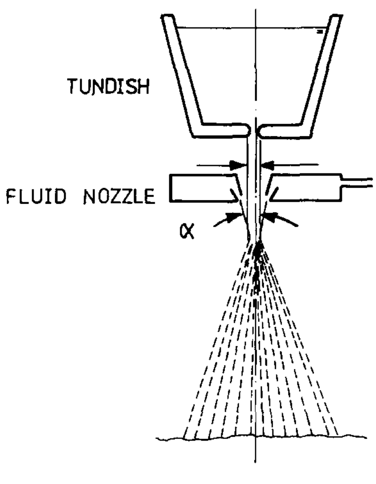

Fig. 1. Powder preparation by atomization.

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and air. Oxidation can be avoided using inert gases.

The effects of many other process variables are widely acknowledged [4–7]. The most important are:

(1) Molten metal properties (chemical composition, surface tension, viscosity and overheat).

(2) Molten metal flow conditions (flow rate, velocity, stream diameter, stream length).

(3) Jet flow (pressure, flow rate, velocity).

(4) Jet geometry (apex angle, position, number of jets).

Nozzle geometry is an important design detail which influences both the jet flow and jet geometry. It is important to know the influence of variables on the quality of the obtained powder. To evaluate such relations it is essential to characterize the powder properly [8]. The main characteristics currently considered are: chemical composition; H_2 loss; particle size distribution; particle shape and the associated properties such as apparent density, flowability, specific surface area; and the microstructure of the particles.

In this paper a 70Ni30Cu alloy has been atomized. Water and air have been used as atomizing fluids at two different pressures to evaluate the effect of the atomizing medium on this particular alloy. The degree of liquid metal overheating was chosen as another process variable to be changed. Powders of varying qualities were thus produced and characterized to assess the effect of the process variables.

2. Experimental procedures

The apparatus used for the atomization is shown schematically in Fig. 2. The jet nozzle for both air and water injection was annular. The apex angles are 30° and 60° for air and water, respectively (Fig. 1).

A 70wt.%Ni–30wt.%Cu alloy was prepared with electrolytic nickel and electrolytic copper. Batches weighing about 8 kg were melted in an induction furnace. The melt heated to the pre-established temperature was poured in the tundish preheated to 900 °C.

The tundish nozzle diameter measured 7 mm. The atomized alloy was collected in a water pool at the bottom of the atomization chamber. After each run, the metal powder was centrifuged and dried in air at 200 °C for 1 h. The yield was determined with the use of a U.S. standard sieve of 210 μm.

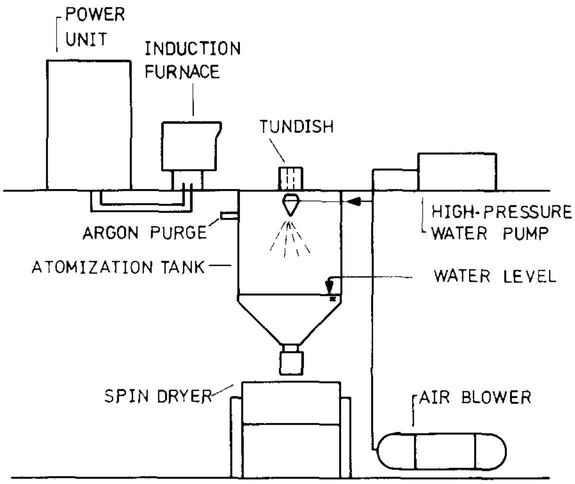

Fig. 2. Experimental atomization unit.

The pouring temperatures were 1550 °C, 1650 °C and 1750 °C with air pressures of 0.2 MPa and 0.3 MPa and water pressures of 7 MPa and 10 MPa.

The powders were characterized by measuring the particle size distribution [9], apparent density [10], flowability [11], and H_2 loss [12]. The particle morphology was also evaluated and metallographic analyses carried out.

3. Results and discussion

The values obtained for the measured properties are presented in Tables 1 and 2 for air and water atomization, respectively. The variation of particle size with cumulative weight percent is shown in Fig. 3 for both processes.

The experimental results are in agreement with similar investigations reported in the literature. The mean particle size is smaller when either the overheating temperature or the atomizing fluid pressure are increased. The variation in particle size was more pronounced with atomization done by air than by water. The effect of overheating is related to the viscosity of the molten metal. Increasing the temperature lowers the viscosity, which leads to easier disintegration of the liquid stream into droplets. Higher pressures increase the energy of the atomizing gas by increasing its velocity. With higher amounts of available energy for liquid metal disintegration, smaller particles are produced.

The photomicrographs of Figs. 4 and 5, taken by scanning electron microscopy (SEM), show the particle shapes produced. Particles obtained by air (Figs. 4(a) and (b)) are more rounded. Higher overheating also improved spheroidization.

Test results of powders with more regular morphology presented higher apparent density and lower flow time (compare the results in Tables 1 and 2). The reason is that more rounded particles have less friction between them so they accommodate or pass one another more easily.

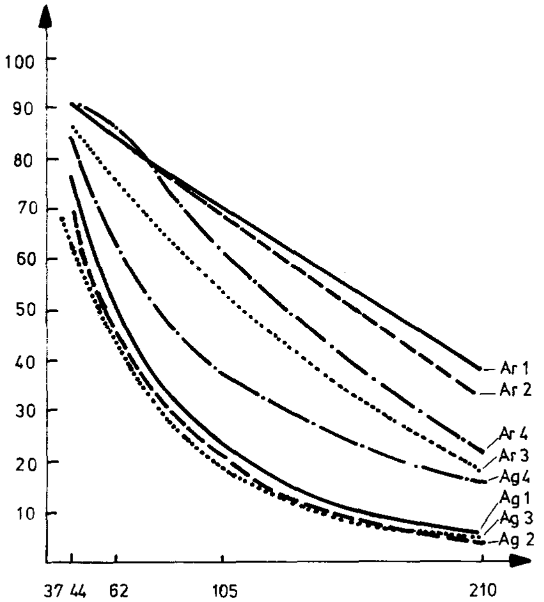

Fig. 3. Cumulative weight percent *vs.* particle size plot for air and water atomization. Ar1, 1550, 0.3; Ag1, 1550, 10.0; Ar2, 1650, 0.3; Ag2, 1650, 10.0; Ar3, 1750, 0.3; Ag3, 1750, 10.0; Ar4, 1750, 0.2; Ag4, 1750, 7.0.

In Fig. 5, particles with more irregular morphology produced by water atomization are shown. Water has a higher heat capacity and so it removes heat more efficiently from the particles, which are also smaller owing to the stronger impact. With no time for spheroidization the particles retain their irregular shapes. It would be expected that an increase of overheat would produce more regularly shaped particles. Nevertheless, comparison of the photomicrographs (Figs. 5(a) and (b)) show no significant morphological differences. These observations are confirmed by the flowability and apparent density test results (Table 2), which do not show significant differences.

The yield increases with both temperature or pressure rise for air atomization. In the case of water atomization only the effect of pressure was confirmed.

Oxidation is more pronounced at higher pouring temperatures and is stronger with air (Tables 1 and 2).

Typical microstructures are shown in Figs. 6(a) and (b). The same type of microstructure was obtained for air and water atomization. Dendritic growth with interdendritic microsegregation can be observed. Such microstructures are usually found in single phase alloys and nickel alloys [13]. The spacing D between secondary dendritic arms was measured as a convenient microstructural parameter. The interdendritic spacing permits determination of the cooling rate ($\dot{T}$) [1, 13].

TABLE 1

Effect of metal overheat and fluid pressure in air atomization

Run no.	Ar1	Ar2	Ar3	Ar4
Pressure (MPa)	0.3	0.3	0.3	0.2
Pouring temperature (°C)	1550	1650	1750	1750
Size distribution (wt.%):				
>210 μm	38.6	32.9	18.0	21.8
210/105 μm	31.9	35.9	35.9	44.6
105/62 μm	13.0	15.2	22.6	19.5
62/44 μm	7.4	7.0	9.8	5.3
<44 μm	9.1	9.0	13.7	8.8
Mean size (μm)	172	160	110	144
Yield (wt.% lower than 210 μm)	61.4	67.1	82.0	78.2
Apparent density (g cm^{-3}) range (210/105 μm)	3.5	4.02	4.06	4.19
Flowability (s/50 g) range (210/105 μm)	25.0	21.3	17.5	17.0
H_2 loss (wt.%) range (210/105 μm)	1.46	2.17	3.24	2.14

TABLE 2

Effect of metal overheat and fluid pressure in water atomization

Run no.	Ag1	Ag2	Ag3	Ag4
Pressure (MPa)	10.0	10.0	10.0	7.0
Pouring temperature (°C)	1550	1650	1750	1750
Size distribution (wt.%):				
> 210 μm	5.9	4.1	5.1	16.3
210/105 μm	18.1	16.7	14.0	22.0
105/62 μm	27.1	24.7	23.6	24.6
62/44 μm	25.8	22.7	25.3	21.1
< 44 μm	23.1	31.8	32.0	16.0
Mean size (μm)	63.0	59.0	51.0	81.0
Yield (wt.% lower than 210 μm)	94.1	95.9	94.9	83.7
Apparent density (g cm^{-3}) range (210/105 μm)	3.35	3.31	3.37	3.62
Flowability (s/50 g) range (210/105 μm)	23.4	25.2	24.3	23.0
H_2 loss (wt.%) range (210/105 μm)	1.35	1.50	1.67	1.57

Fig. 4. SEM for air atomized powder in the range 210/105 μm. (a) 1550 °C; 0.3 MPa. (b) 1750 °C; 0.2 MPa.

There is experimental evidence indicating that

$$D = K\dot{T}^{-n}$$

where K and n are constants.

The interdendritic spacings measured over two granulometric ranges are given in Table 3. These results show that the finer the particle, the smaller are the interdendritic spacings, which would be expected on the basis that cooling rates should be higher. Similarly, the interdendritic spacings found for the water atomized powders are smaller compared with those obtained by air atomization at the same pouring temperatures, owing to the higher cooling rates promoted by water.

Comparison of the results obtained with the two different atomization fluids indicates that water produces both a smaller mean particle size and less oxidation. Powders produced by air atomization present higher apparent densities.

It can be concluded that an inert gas with higher pressures (above 0.3 MPa) could be a

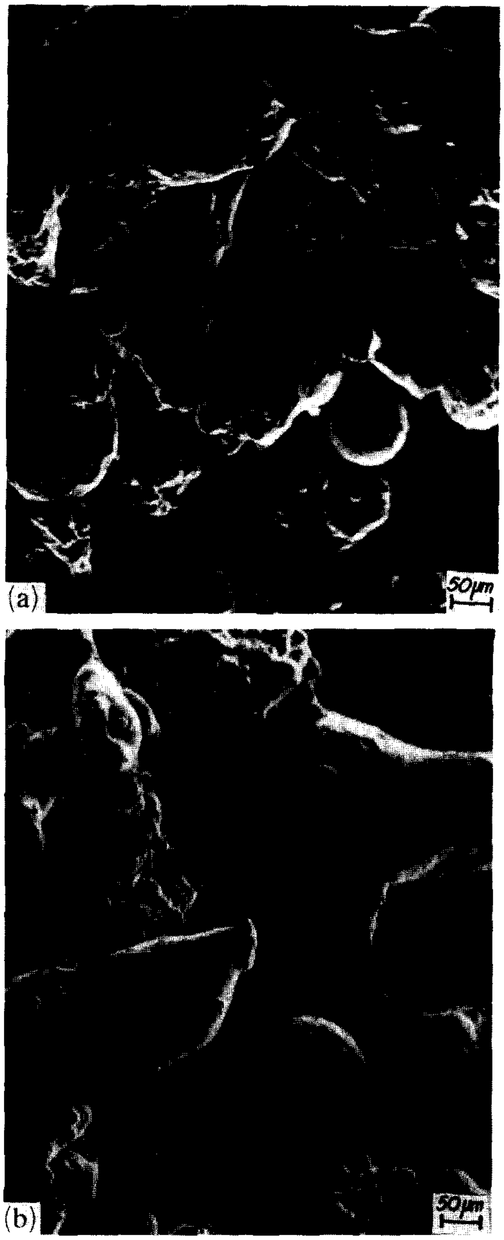

Fig. 5. SEM for water atomized powder in the range 210/105 μm. (a) 1550 °C; 10.0 MPa. (b) 1750 °C; 7.0 MPa.

reasonable alternative to produce powders with smaller mean particle size and reduced H_2 loss, and higher apparent density.

4. Conclusions

(1) The mean powder particle size was smaller for higher overheating by both air and water atomization.

(2) The mean particle size was smaller for higher atomization fluid pressure.

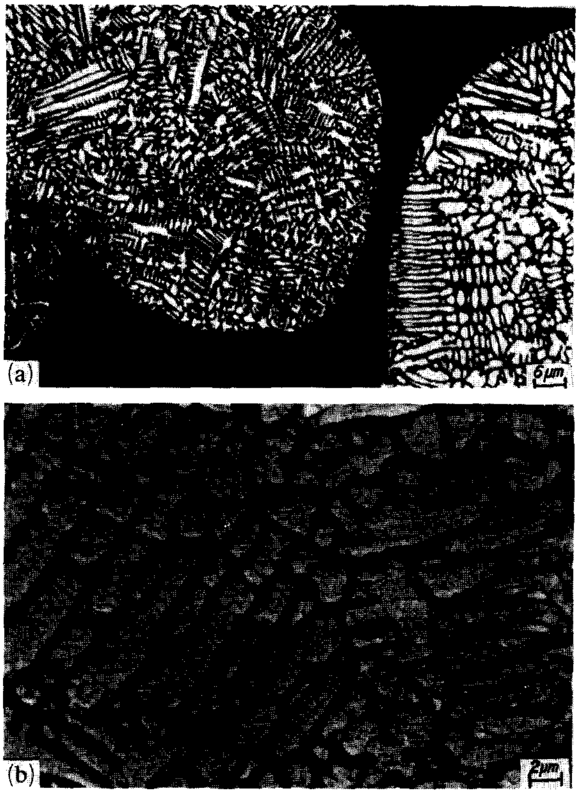

Fig. 6. Optical micrographs for air and water atomization.

TABLE 3

Interdendritic spacings for air and water atomization (μm)

Size range	Air	Water
105/62 μm	1.85 ± 0.36[a]	1.34 ± 0.29[a]
62/44 μm	1.28 ± 0.27[a]	0.95 ± 0.18[a]

[a]First number: mean value; second number: standard deviation.

(3) The particle shape tends to be more spherical with air atomization and tends to be more irregular with water atomization. Accordingly, powders with rounded particles produced by air atomization give higher apparent densities.

(4) The interdendritic spacing is smaller for water atomization, confirming that air produces a slower cooling rate.

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