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RAPID SOLIDIFICATION OF A MODIFIED 7075 ALUMINUM ALLOY BY ULTRASONIC GAS ATOMIZATION

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ABSTRACT

Variables associated with ultrasonic gas atomization of aluminum alloys to attain rapid solidification at rates of about 10^4 to 10^5 K/s are discussed. Both structures and properties of some of the alloys produced are shown.

Introduction

The slow solidification of large ingots of high alloy content aluminum alloys, coupled with normal impurity levels of Fe, Si, Mn, and other elements, leads to coarse grained, segregated structures typically containing intermetallic inclusions up to 50 microns across. These coarse inclusions are recognized to be prime sources of fatigue crack initiation and generally result in poor toughness properties.

Rapid solidification (typically 10^3 to 10^6 K/s) of compositions has demonstrated that the brittle intermetallic phases can be totally suppressed; the resultant hot densified alloys can be produced as fine grained structures, with rare second phase particles larger than about one micron in size. Work demonstrating these effects has been reported for rapidly solidified (RS) splatted 2024 alloy[1], roll quenched modified 7075[2], and roll quenched 2024 alloys with 1 and 3% lithium[3].

More recently RS aluminum alloys have been produced by an ultrasonic gas atomization process based on a Swedish development[4] which applied a Hartmann shockwave tube to produce a pulsed, high velocity gas jet to accomplish the atomization. As has been demonstrated by Johnston and See[5], high gas velocity is a critical parameter in atomization. The shear resistance of a liquid metal stream is low; by speeding up the velocity of the liquid stream, disintegration takes place progressively to produce finer particles which should become spheres in the absence of refractory oxide films on the surface of the liquid.

It has further been shown[6] that to achieve maximum values of h, the heat transfer coefficient, one strives to produce fine liquid spheres and high particle velocity in the cooling fluid (gas or liquid).

Gas velocities, and therefore the velocity of the atomized liquid spheres, are generally less than about Mach 0.5 in conventional gas atomization processes[6]. According to the Swedish work, the Tove-Nilsson atomization die[4], by virtue of its Hartmann tubes, accelerates the atomizing gas to velocities in excess of Mach 2. Figure 1 shows a typical shock wave pattern from a Hartmann tube for one selected geometry and gas pressure. Purely on the basis of such high gas velocities, finer powder particles would be expected according to Johnston and See[5]. The superimposition of a pulsed gas stream leads to more efficient atomization.

Experimental Procedures

A series of circular steel dies were prepared incorporating the Tove-Nilsson designs[4]. The die is a hollow cylinder of about 32 mm (1.25") OD, 12.55 mm (0.5") ID and 25 mm (1.0") high. A circular manifold distributes high pressure gas to, typically, 16 to 24 jet nozzles. One or more gas inlets may be used to distribute the gas uniformly in the manifold. Gas pressures up to 8.1 MPa (1200 psig) are used, depending on the desired atomization product (average size and size range), and on particular alloy characteristics (surface oxide films, for example, tend to produce a coarser powder size).

The atomization die is water cooled to minimize distortion due to uneven heating from the metallic stream, and to give partial protection in case of a metal stream break out from the delivery tube.

The Hartmann shock-wave tube design, based on Swedish measurements, shows a broad frequency band (crystal pick-up) near 80,000 cps, and a minor broad band near 20,000 cps (see Fig. 2).

The gas jets from the nozzles converge near the center point in the metallic stream and form an included angle of 45°. Angles smaller than about 45° tend

to be less efficient and may develop turbulence; the reflection of the gas stream off the metallic stream is greater the smaller the included angle. Converging gas stream angles significantly larger than 45°, while more efficient (less gas stream reflection), generate a higher back pressure which can result in stoppage of liquid metal flow and even gas bubbling up through the metal bath in the crucible or tun dish.

One very important characteristic of the gas jets is the very large cooling effect due to gas expansion on exiting the die. This can create problems in metal flow if sufficient metallic superheat is not provided.

Overall, the dimensions of the die and distances between fixed points are very important, and geometries are critical. Short gas paths are recommended to avoid turbulent flow. The distance from the tip of the ceramic metal-delivery tube above the gas jet is important in terms of gas back pressure and in terms of metal flow: the very high velocity of the gas stream aspirates the liquid metal and sharply increases its velocity, thinning the resultant metallic stream.

Figure 3 shows our current tank and melting set-up for small melts (under 5 lbs). A 15 KW M-G induction melting unit permits rapid melting plus stirring of the bath to assure a homogeneous melt. Melting is done under argon, after the tank has been evacuated and back-filled with argon twice (when clean atmospheres are necessary). Nitrogen is used where gas-metal reactions are not of concern. Small melts are bottom poured, using a graphite stopper rod (usually). For larger melts, tilt pouring, using a tun dish, is preferred.

The mild steel, welded tank is 4 ft. in diameter (1.22 m) with the lower, atomization section about 5 ft. long (about 1.5 m). Since a large amount of gas is delivered during the short atomization period, pressure build-up is averted through use of relief valves. The significant quantity of fines (less than about 20 microns) are collected in a cyclone separator.

The relatively small size of the tank is ideal for laboratory work, and is possible because there are rare powder particles as coarse as 500 microns. Figure 4 shows the relationship between powder size and distance traveled (or time of flight) for gravity fall to assure greater than, say, 90% solidification. Accepting the much higher particle velocities associated with the USGA process, plus an unknown contribution from the chilling effect of the expanding gas exiting the atomization die, solidification is, in fact, much faster. Our experience shows that we have not experienced problems of our largest particles remaining liquid till reaching the collected solidified finer powders at the foot of the tank where they would be fused together by the arriving liquid droplet. We did in fact originally work with a 3 foot diameter tank which was 4 feet high (0.91 m round by 1.22 m tall) and also did not experience problems with unsolidified larger droplets.

Of interest, when atomization is first started into an evacuated tank, the same fine atomized liquid droplets (argon atomization) reach the container walls in the liquid state and are splatted against the walls.

Some Results

Figure 5 shows two views of finer and coarser fractions of USGA aluminum alloys. Particles are uniformly spherical; in flight collisions are infrequent as indicated by the relatively infrequent attached fine particles. Examination of many hundreds of sectioned powder particles shows rare instances of hollow particles. The absence of such gas cavities supports arguments that the liquid particles leave the metallic stream as compact shapes rather than as flakes, platelets or elongated rod-like structures. For these reasons we assume that the liquid spheres are formed in a single

step process rather than in the three-step process described by Johnston and See[5] from their high speed movies of low gas velocity atomization.

Figure 6 shows the range of powder sizes obtained in typical atomization runs for aluminum. Gas pressures (gas velocities) must be high to achieve high yields of minus 44 micron (325 mesh) powders, and dies must be clean, with smooth cavities, etc. Typically the degree of superheat is very important. The values of 770°C shown here are in fact low compared to commercial practices for aluminum where superheats up to 850 to 900°C are not uncommon.

With USGA it is our practice to use the lowest superheat possible while striving to achieve the desired solidification rate as measured in terms of secondary dendrite arm spacing (DAS).

Of prime interest is the quench rate from the melt. Atomization is therefore calibrated to achieve certain maximum solidification rates as measured by DAS. In turn, if all heating steps are kept to selected minimum values, including hot consolidation, the fine DAS tells us, we believe, what to expect in terms of supersaturated solid solutions, segregation phase formation and second phase particle size, and will also indicate the potential for production of a fine grain size.

Figure 7 shows the variation of DAS for aluminum alloy powders up to 250 microns. For most of the work to date, we have accepted 250 μ because, while obtaining useful high yield of powders, we are able to obtain the degree of refinement of structure we desire, with uniform microstructure in the final hot worked and heat treated product. Actually we would prefer to eliminate the -10 micron fraction to minimize the amount of oxide in the final product. Based on Fig. 7 and solidification rate data for aluminum, we estimate solidification rates of 10^4 to 10^5 K/s for the -250 micron powders.

Powder Consolidation. Because we melt under argon and atomize with argon, we are able to avoid the usual high temperature powder treatment to break up the hydrates on the surfaces of air atomized aluminum powders. Our powders are exposed to air briefly on removal from the atomization tank and again when packing and cold compacting into cans (the powders are stored in a dry box in the meantime).

We avoid the 500°C plus treatments to break down the hydrates because we have seen evidence of structural coarsening in some of our rapidly solidified alloys at 500°C and above.

There have been no instances of blister formation in our hot extruded alloys even at solution temperatures as high as 570°C.

The cold compacted aluminum cans are welded, heated and evacuated at nominal vacuum levels. Heating is typically to 300 to 400°C, which is also the extrusion temperature (400° more commonly than 300°C). The extrusion ratio is generally 30:1 (area), and both round bars and rectangular bar sections (0.5 X 1.5 inch) (1.27 X 2.80 cm) are made depending on test requirements. Neither hot mechanical nor hot isostatic pressing are used although there could clearly be advantages if an inexpensive facility was available capable of hot charging and discharging the aluminum cans.

Table I shows some typical tensile values for 2000 series aluminum alloys (2024 and 2020) at room temperature. Similarly, Table II shows values for the 7000 series alloys at room temperature. Fairly minor compositional variations are evident in Table I and II, with important increases in strength (and other properties). The areas of alloy and structure development are clearly wide open for significant advances.

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TABLE I

Room Temperature Tensile Properties of 2000 Series Alloys

Alloy	Heat Treatment	DAS (μ)	σ_{yp} (ksi)	σ_{uts} (ksi)	El %	RA %	Authors
2024 - T351 Extrusions	S.T. 495°C Age: R.T.	—	52	71	10	—	—
2024 Splat cooled 50% cold swaging	S.T. 495°C Age: R.T.	0.7	47	79	24	39	Lebo et al[1]
2024 + 1% Li Roller Quench + Ext	S.T. 495°C Age: R.T.	∿1	56	76	21	10	Sankaran et al[3]
2024 - T6	S.T. 495°C Age: 190°C/12 hr	—	56	68	5	—	—
2024 - 1% Li Roller Quench + Ext	S.T. 495°C Age: 190°C/12 hr	∿1	63	77	8	6	Sankaran et al[3]
2024 + 3% Li Roller Quench + Ext	S.T. 495°C Age: 165°C/12 hr	∿1	83	84	5	3	Sankaran et al[3]
2020 + 1.5% Li USGA + Ext	S.T. 530°C Age: 210°C/4 hr + 190°C/16 hr	∿2	88	94	7	12	S. Kang and N. J. Grant

TABLE II

Room Temperature Tensile Properties of 7000 Series Alloys

Alloy	Heat Treatment	DAS (μ)	σ_{yp} (ksi)	σ_{uts} (ksi)	El %	RA %	Authors
7075 Extrusions	S.T. 460°C Age: 120°C/24 hr	—	78	87	7	49	Durand et al[2]
7075 Roller Quench + Ext	S.T. 460°C Age: 120°C/24 hr	1	72	87	9	19	Durand et al[2]
7075 + 2% Zn Roller Quench + Ext	S.T. 460°C Age: 120°C/24 hr	1	89	96	4	4	Durand et al[2]
7075 + 1%Fe + 1%Ni Roller Quench + Ext	S.T. 475°C Age: 120°C/24 hr	1	92	104	9	20	Durand et al[2]
7075 + 1.6Fe + 0.6Ni USGA + Ext	S.T. 490°C Age: 120°C/24 hr	1.5	83	100	6	12	Anand and Grant
CT91 Air Atomized + Ext	2 step heat treatment	—	85	89	—	—	Alcoa
CT90 Air Atomized + Ext	—	—	93	97	—	—	Alcoa

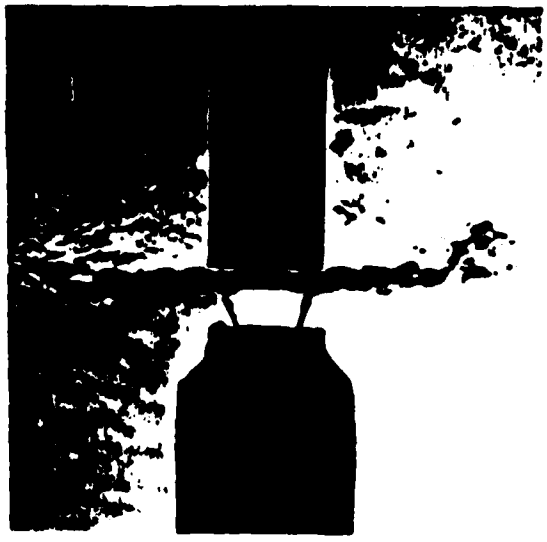
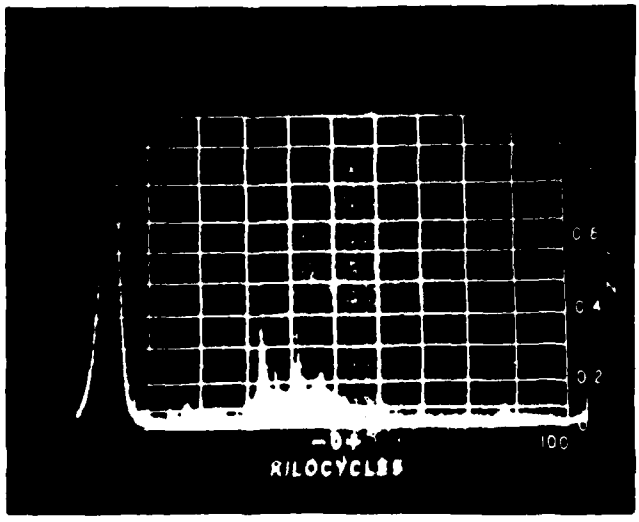


Figure 1. Hartmann tube with typical shock wave pattern for a given geometry and gas pressure.[4]



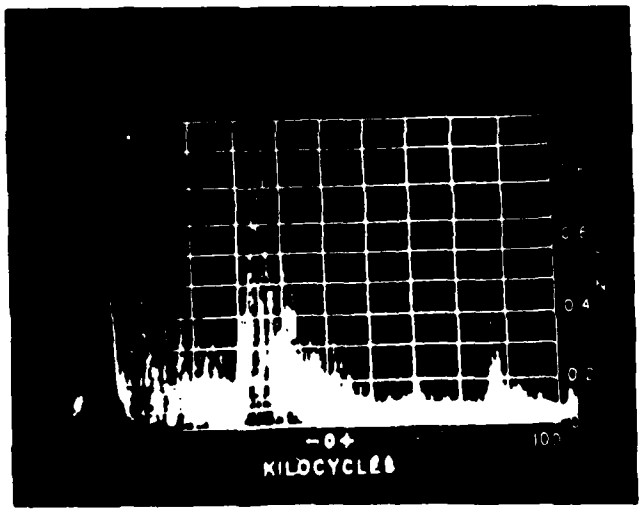


Figure 2. Typical accoustic patterns established by crystal pick-up on a Tove-Nilsson die at 10 and 30 atmospheres.[4]

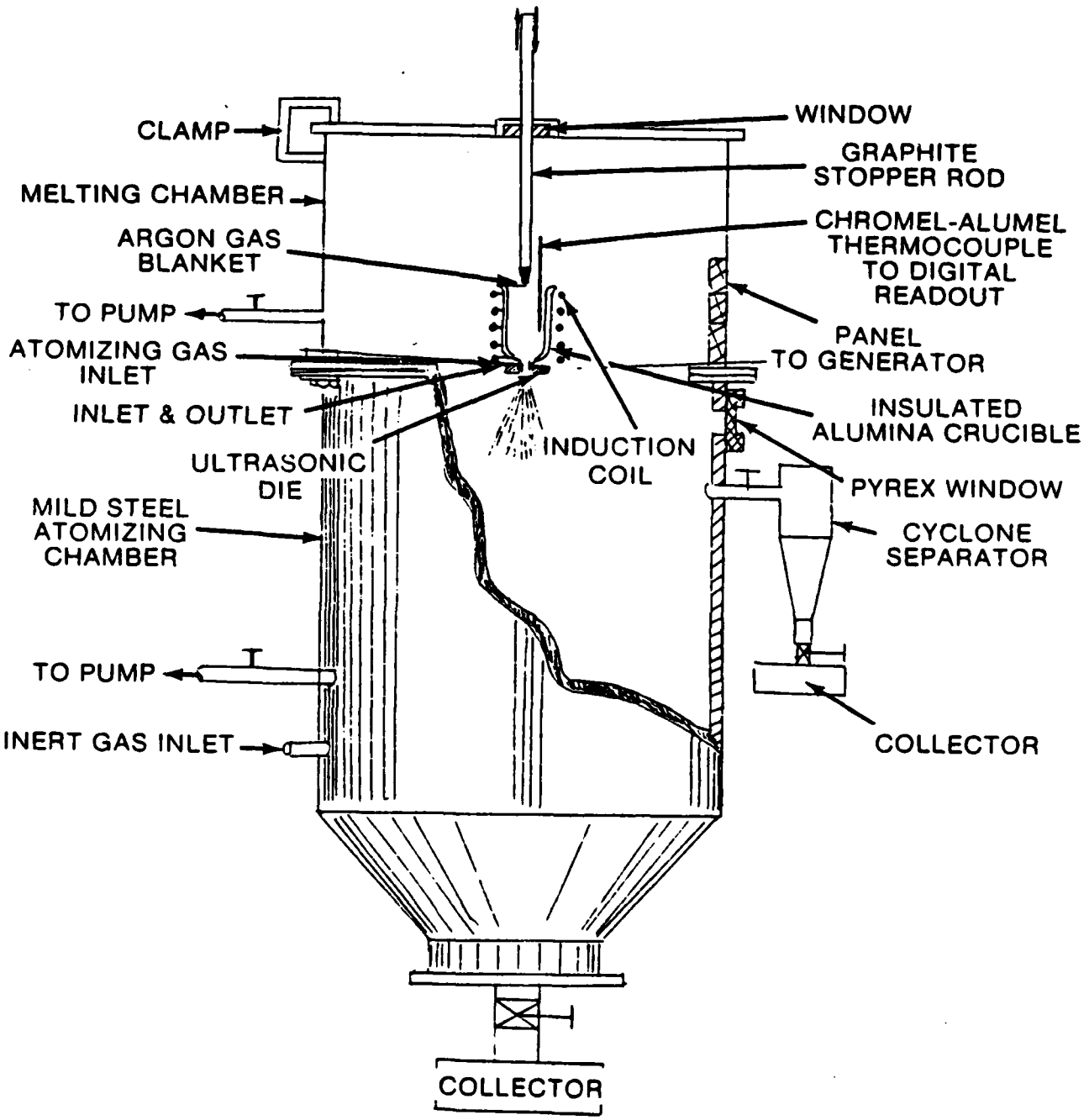


Figure 3. Schematic view of atomization chamber.

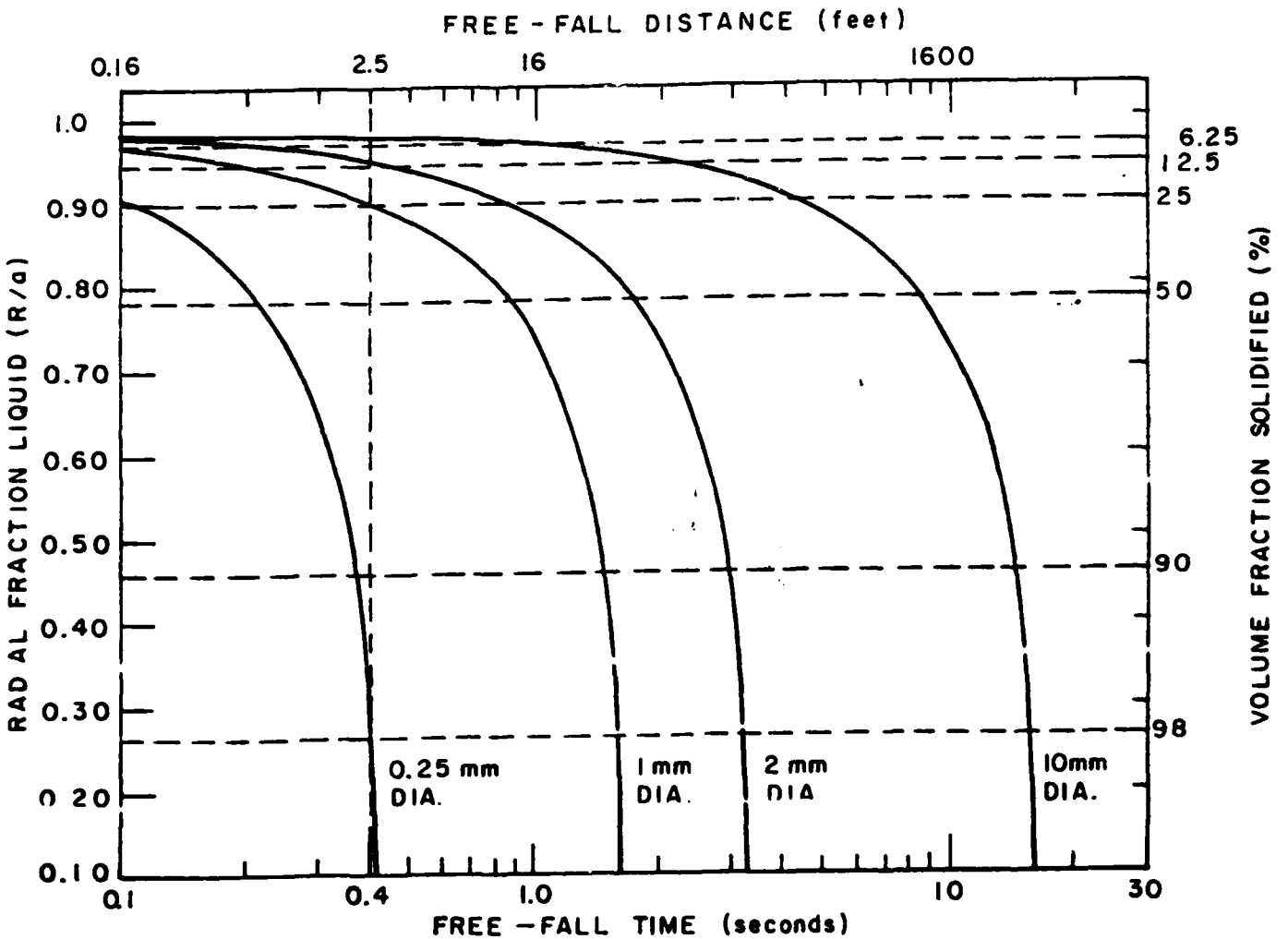


Figure 4. Radial fraction liquid (or vol % solid) versus free-fall time or distance for spherical liquid droplets of steel.

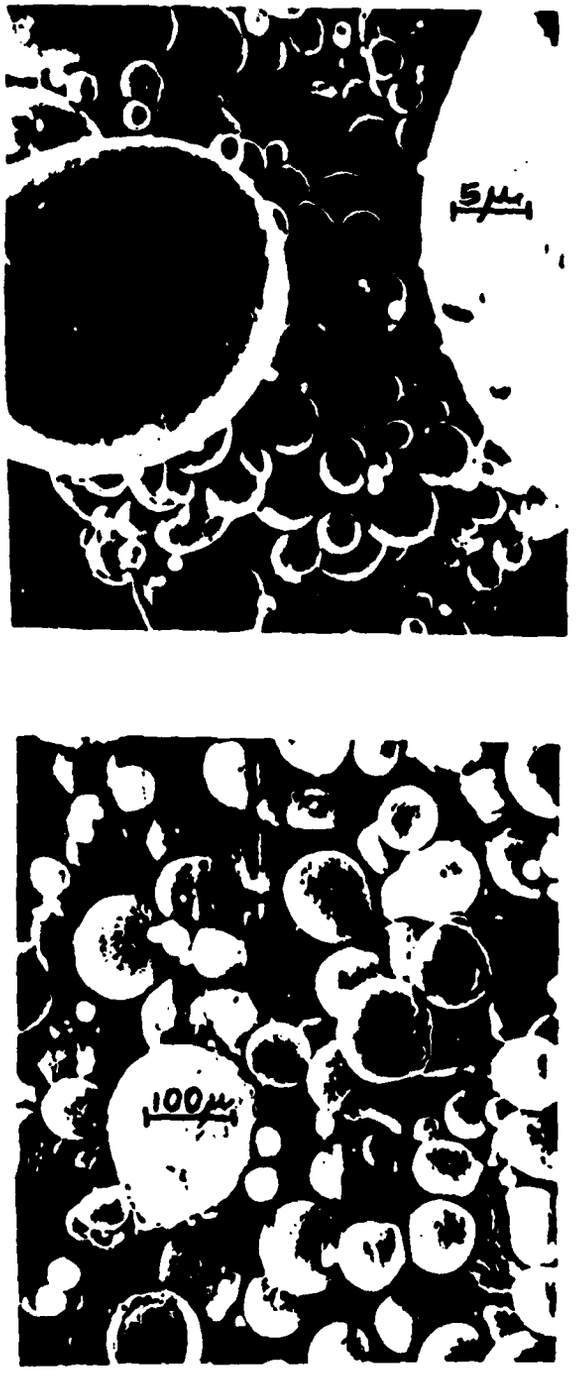


Figure 5. Two views of USCA aluminum alloys.

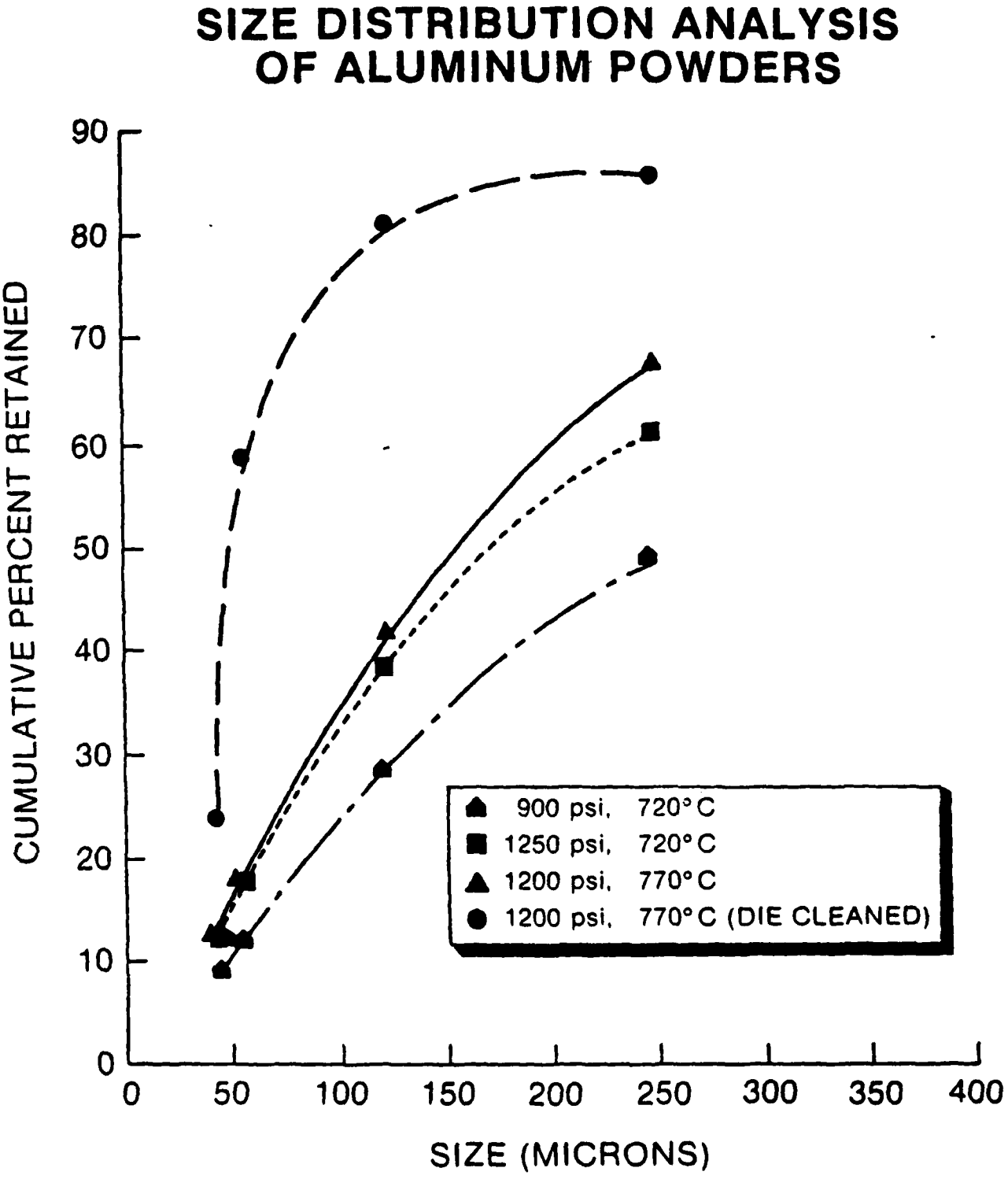


Figure 6. Powder size ranges for USGA aluminum as a function of superheat temperature and gas pressure (velocity).

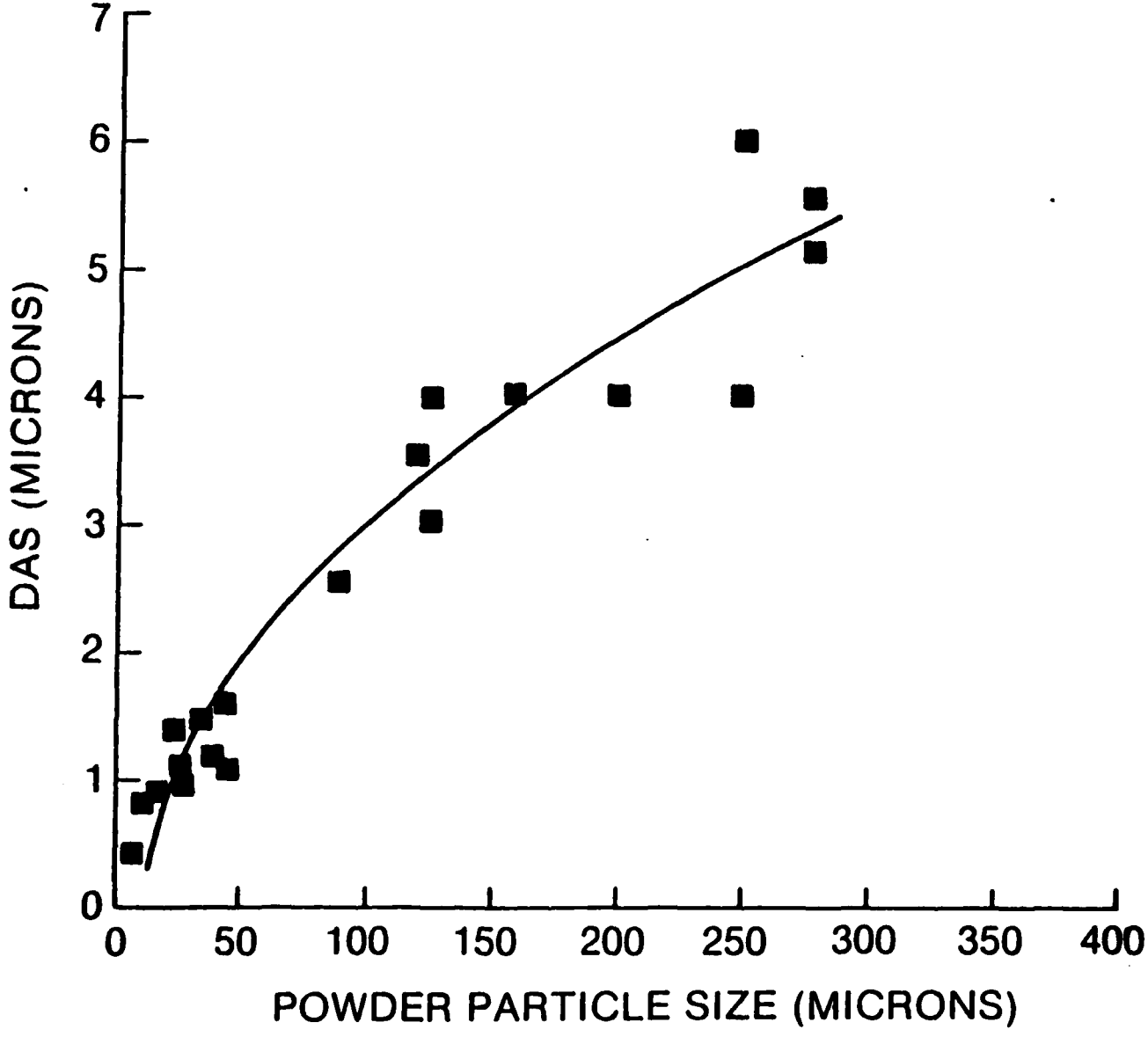


Figure 7. Secondary dendrite arm spacing versus powder particle size for alloyed aluminum powders made by USGA.