

Fig. 13 Particle size distribution measured by Microtrac in some Alcoa powder grades (grade 130/2 is by sonic sieve method)



Fig. 14 Variation of mass median diameter for aluminum powders with the ratio of metal/gas

slowed down in order to make finer powders. The median diameter is found to be approximately proportional to the square root of the rate of atomization. This may be more generally expressed as a function of the metal/gas flow rate ratio (Fig. 14). Superheat in the molten metal was found to have only a small effect on the powder size providing that there was sufficient heat in the liquid to prevent premature solidification. This is reasonable, as any change would come about primarily through changes in such properties of liquid metal as surface tension, density, and viscosity. These properties are only mildly dependent on temperature.

As metal flowrate is increased and the powder becomes coarser for any given atomizing conditions, in general, the spread in powder sizes (as measured by  $\sigma_g$ ) also increases (Fig. 15). This spread has been taken as indirect evidence for the presence of stable particle sizes in the powder after the completion of secondary breakup (Ref 5, 7, 8, 21).



Fig. 15 Comparison of argon and helium gases on the basis of gas volume used per unit weight of powder produced

### **Particle Morphology**

The morphology of atomized aluminum powders is influenced strongly by the amount of oxygen present in the gas phase during atomization. Particles atomized in an inert gas show spherical features, whereas air-atomized particles are of irregular shape (also known as

"nodular" powders). This effect of oxygen on morphology has been attributed to the pinning effect of the oxide nuclei that form on the droplets before they have solidified (Ref 23). Many such nuclei form rapidly on a droplet when there is a high level of oxygen in the gas phase leading to effective pinning at those points. Whereas at low oxygen levels or in inert gas, the rate of oxidation would be slow and not cause pinning in which case surface tension forces would be able to maintain the spherical shape of the droplet until solidification. Tests with gaseous atmospheres of varying oxygen content have shown that the change in morphology takes place rather sharply at an oxygen level between 2 and 4%. Powders made in a gaseous atmosphere with 2% oxygen or less show essentially the same degree of sphericity as inert gasatomized powders. When oxygen is above 4%. the deviation sphericity approaches those levels observed in air-atomized powders. The change in shape of the particles as a function of oxygen level in gas is attributed to the distortion of the droplets by oxide formation on the surface. Brunauer-Emmet-Teller (BET) surface area measurements indicate that the change in morphology takes place at an oxygen level of  $\sim 2\%$  in the case of pure aluminum powders (Fig. 16).

Particle size and superheat are also known to influence the degree of sphericity of powder particles. Smaller particles tend to be closer to spherical than larger particles made under the same conditions.

Another feature of atomized aluminum powders, particularly important in milling applications, is the surface quality of the particles. Finer powders tend to have excellent surface quality. In others, cells can be seen.

Satellites form by warm/cold welding of smaller particles onto large particles during atomization. If the collision takes place before the larger particle has solidified, the satellite particle will be embedded.

**Specific Surface Area.** The specific surface area of aluminum powders is determined by the size distribution and the morphology of the particles. Powders atomized in air show a larger specific surface than inert gas-atomized powders due to the irregular shape. Figure 17 shows specific surface area of aluminum powders measured by the method of gas adsorption (BET). Fine air-atomized powders can have specific surface area values approaching  $1\text{m}^2/\text{g}$ . Medium sized powders are typically in the 0.2 to 0.4 m<sup>2</sup>/g. For spherical powders at any given mass median diameter, the specific surface area values are values are values area values area values area values area values area bout one-half of that of air-atomized powders.

It is possible to estimate the surface area (S) of atomized powders from the distribution of particle sizes. This is related to the Sauter mean diameter  $d_{vs}$  that was defined above. For powder particles of perfect spheres, the relationship is:

$$S = \rho d_{\rm vs}$$



Fig. 16 Influence of oxygen content of the gaseous medium on BET surface area of aluminum powders



Fig. 17 BET surface area for aluminum powders atomized in air and in inert gas

Because atomized aluminum powders are not geometrically perfect spheres, they show a greater surface area. For inert gas-atomized powders, a factor of 1.3 leads to good estimates (Ref 21). A greater factor in the range of 2.6 to 3.5 is needed for the irregularly shaped air-atomized powders.

#### Surface Oxide Content

Aluminum reacts readily with moisture or free oxygen in the air during manufacture to form an oxide coating on the powder surface. The amount of surface oxide formed during atomization is strongly dependent on the oxygen level of the atomizing atmosphere and particle size of the powder produced. The oxygen content of fine powders atomized in air can approach 1% level by weight (Fig. 18). For coarser powders, 0.4% oxygen is more typical. A mean oxide thickness can be calculated from the oxygen content of the powder if the specific surface area has also been determined. It is then found that the thickness of oxide is, in fact, greater for coarser powders despite the lower overall oxygen content (Fig. 19). Finest powders under 10  $\mu$ m show a mean oxide thickness of 4 to 5 nm, whereas with coarser powders, the thickness can reach 10 nm.

Spherical powders, atomized in an inert gas, contain substantially less oxygen compared to air-atomized powders. This is in part due to the smaller specific surface area of these powders. However, when compared to the mean oxide thickness, the oxide is actually between 2 and 5 nm thinner with relatively little dependence on the size of the powder.

While the mean oxide thickness is useful for comparison between different powders, it does not provide any information on the nature of the oxide skin. Indeed, recent observations have indicated that the oxide on aluminum powders is not a uniform thickness (Fig. 20). Most of the surface is covered with a thin oxide film with interspersed high hills up to 20 nm thickness (Fig. 21). The high hills represent the oxide that forms at discrete nucleation sites while the droplet is still in the molten state. As noted above, the distortion of the droplets by the nuclei is considered to be the cause of the irregular morphology of the airatomized powders. The space between the mounts is covered by a thin skin that mostly forms in the solid state. A similar description has been offered for alloyed powders. The greater mean thickness observed in coarser air-atomized powders is consistent with this mechanism because they will have spent a longer period in the molten state exposed to oxygen.

Temperature of the metal would obviously have a bearing on the amount of oxide formed in powders. The results relate to material obtained in regular atomizing conditions with a superheat of 150 to 200  $^{\circ}$ C. If higher temperatures were to be employed, higher oxide levels would be expected.

The surface of the aluminum powder is hygroscopic and will react with moisture to form Al(OH)<sub>3</sub> by hydration of the oxide and corrosion of the metallic aluminum especially in fine powders. A minimum relative humidity of 60% is needed for hydration promoted by prolonged exposure to moisture and temperature. The presence of manganese and chromium has been found to sharply decrease the formation of Al(OH)<sub>3</sub> in alloyed powders.

#### **Chemical and Physical Properties**

The chemical compositions of unalloyed atomized aluminum powders are shown in Table 1. Iron and silicon are the major contaminants for both regular and high-purity powders. In many cases, atomizers have access to molten metal from a nearby aluminum smelter for regular purity metal grades. For high-purity powder, metal is typically bought in the form of purified ingots (sows) and prepared in furnaces that are suitably lined.

High-purity aluminum is resistant to attack by acids, but it dissolves in a mixture of nitric and hydrochloric acids. Solutions of alkali



Fig. 18 Oxygen content for aluminum powders



Fig. 19 Mean oxide thickness for aluminum powders

hydroxides rapidly attack aluminum, with evolution of hydrogen and formation of the corresponding soluble aluminate. Aluminum reacts vigorously with bromine and iodine to form aluminum halides. Dry, oxygen-free chlorine or hydrogen chloride gas also reacts with aluminum to form aluminum chloride. Finely divided aluminum can react violently when exposed to halogenated hydrocarbons, such as methylene chloride and carbon tetrachloride, especially if the operation is carried out under pressure. Aluminum is stable in air because of its thin, natural oxide film. In finely divided powder form, however, aluminum is more chemically reactive and hydrates when exposed to moisture as described above. Powders also react with water to liberate hydrogen and form aluminum hydroxide and release heat in the process.

**Physical Properties.** The physical properties of aluminum and pure aluminum powders are listed in Table 2. The real density of aluminum powder approaches that of the base metal, but



Fig. 20 Simplified model of nonuniform oxide layer consisting of thick islands on a thin skin



Fig. 21 Model of oxide formed on gas-atomized Al5-Mn6Cr powder particle. (a) In the as-atomized state. (b) After exposure to a humid atmosphere. Source: Ref 24

both apparent density and tap density vary as a function of particle size distribution.

#### **Explosion Potential**

Many materials, including metals, polymers, and composites, are potentially explosive in the powdered form. These dusts may be the product of a given process or an unwanted byproduct. Safe handling of potentially explosive powders requires recognition of the hazards, proper handling techniques, and awareness of explosion prevention methods and fire fighting techniques.

The typical explosion occurs due to the simultaneous occurrence of the following:

- A dust cloud
- An oxygen-containing agent
- An ignition source

Common fuels include flammable gases, degradation by products, plastics and other carbonaceous particles, vapors deposited during alloying, or other molten metal handling operations, and aluminum or other metal dust cloud or layer. Common oxidizers include oxygen containing polymers, salt oxides, metal oxides (like  $Fe_2O_3$ ), carbon dioxide, water, and, of course, air. Typical igniters include hot surfaces, electric or static arcs, spontaneous combustion, pyrophoric materials, impact sparks, thermite reactions, and matches and lighters.

The probability of explosion for different materials is ranked by the United States Bureau of Mines as severe, strong, moderate, weak, and none. These ratings only describe the probability of an explosion occurring as a function of the minimum amount of airborne material required to initiate and sustain a catastrophic reaction. These ratings do not describe the force of there action.

Aluminum, magnesium, and lithium have severe ratings. They are aggressive reducing agents whose reactions occur with a drastic release of energy. On the other hand, copper, stainless steel, and nickel do not oxidize readily and therefore are low on the explosibility list. In Table 3, we compare these metal powders to nonmetallic materials.

The rate of pressure rise is the main property that separates metal powders like aluminum and magnesium from organic materials like plastics and flour. If corn flour can destroy a silo at 3,700 psi/s, imagine what a metal dust at 5 to 6 times that will do!

Explosibility is particle size dependent, as noted in Fig. 22. One of the first rules used to predict explosibility potential is to characterize the amount of material finer than 200 mesh (75  $\mu$ m) in a sample. Two things become apparent. First, below 10% –200 mesh, aluminum powders have typically shown no explosion hazards. Second, above the 10% level, the minimum amount required drops exponentially to a strong hazard, above 40%, the explosion probability is at the severe level.

**Preventive measures** must be geared at controlling and/or eliminating as many of the legs of the triangle as possible: the fuel, the oxidizer, and/or the igniter. Table 4 shows several properties of aluminum that need to be taken into consideration when establishing preventive measures.

Prevent formation of dust clouds when handling explosive powders. Housekeeping is the most effective prevention method. Using plant air to blow away dirt is prohibited. Natural hair brushes should be used to clean up work areas. Metal dust pans or containers are preferred.

*Eliminate/Control Ignition Sources.* The most obvious sources include: hot surfaces, smoking, and electrical grounding of equipment. More rigorous items include: The need for special nonsparking hand tooling, dust-tight/explosion-proof electrical switches, connectors, and other equipment, selective use of conductive floors and attention to welding and torching operations.

In the event of a fire or explosion, some simple rules for metal dusts are:

#### Table 1 Typical chemical analyses of atomized aluminum powders

		Composition, wt%		Other metallics	
Type of powder	Aluminum	Iron	Silicon	Each	Total
Atomized powders					
Typical	99.7				
Maximum		0.25(a)	0.15(a)	0.05	0.15(a)
High-purity atomize	d powders				
Minimum	99.97				
Typical	99.976	0.007	0.008		0.009
(a) Iron plus silicon, 0.30	) wt% max				

## Table 2Typical physical properties ofatomized aluminum powders

Wrought density (metal), kg/m <sup>3</sup>	2700
Melting point, °C (°F)	660 (1220)
Boiling point, °C (°F)	2430 (4410)
Surface tension at 800 °C (1470 °F), N/m	0.865
Apparent density, kg/m <sup>3</sup>	800-1300
Γap density, kg/m <sup>3</sup>	1200-1500
Melting point of oxide, °C (°F)	2045 (3720)
Dxygen content, wt%	0.1 - 1.0
Source: Ref 5	

#### kg/m³ 1.000 0.800 None explosive limit, rating 0.600 Explosibility Weak 0.400 Moderate 0.200 Lower Strong ٥ Severe 20 40 60 80 100 Particle size (-200 mesh), %

**Fig. 22** Effect of aluminum powder particle size on explosibility. Shown by comparing lower explosion limit with the percentage of particles less than 200 mesh in size

- Know and recognize the hazard
- Evacuate and let the event run its course
- For small, localized fires, initiate alarm procedure first and then only use Class D fire extinguishers or dry inert granular material (for example, sand) to smother the fire.
- Do not use water. Water reacts with aluminum and many other finely divided metals to form hydrogen, which is an even worse problem, and water usually comes out of the hose under pressure. This in turn creates a dust cloud that can turn a simple metal fire into a dust explosion.

Your local fire department should be aware of the hazards around metal fires and prepared in proper fire fighting methods.

**Selected References.** More information on the topic of powder handling and the hazards involved for aluminum powders include:

## Table 3 Relative explosibility of various dusts

Material	Lower explosive limit, oz/ft <sup>3</sup>	Rate of pressure rise, psi		
Aluminum	0.045	20,000		
Magnesium	0.030	15,000		
Polyethylene	0.020	7500		
Flour	0.050	3700		
Coal	0.050	2000		
Coffee	0.085	150		
	0.005	150		

 $1 \text{ oz/ } \text{ft}^3 = 1 \text{ kg/m}^3$ . 1 psi/ s = 6.9 kPa/ s

## Table 4Explosibility characteristicsof aluminum powders

Minimum concentration (lower explosive	0.045 (0.045)
limit), kg/m <sup>3</sup> (oz/ft <sup>3</sup> )	
Minimum igniting energy, mJ (cal)	50 (0.012)
Ignition temperature of cloud, °C (°F)	650 (1200)
Ignition temperature of layer, °C (°F)	760 (1400)
Maximum explosion pressure (gage),	503 (73)
kPa (psig)	
Maximum rate of pressure rise, MPa (psi/s)	140 (20,000 +)

- "Recommendations for Storage and Handling of Aluminum Powders and Paste," Publication TR-2 by the Aluminum Association, Inc.
- "Explosibility of Metal Powders," Bureau of Mines Report 6516, United States Department of Interior, 1964
- Material safety data sheets

#### Applications

Aluminum powders have physical and metallurgical characteristics related to their method of manufacture that make them extremely important in a variety of applications. They can propel rockets, improve personal hygiene, make computers more reliable, refine exotic alloys, and make the family sedan or the newest Air Force fighter lighter in weight and more efficient while providing beauty and protection against sunlight and moisture. Powders, in the form of PM parts for structural and nonstructural applications, also hold the key to some of the most exciting new developments in the future of aluminum.

As powder, aluminum is used in blasting agents where the concentrated energy from the high heat of combustion allows explosive manufacturers to better match energy release to specific rock characteristics or mining conditions. This eliminates or minimizes over or under blasting and reduces total rock breakage costs. This same energy release characteristic makes powders an important element in the solid fuel rockets used for national defense and to launch space probes. Each launch of the Space Shuttle requires 350,000 pounds of aluminum powder. Energy release is also an important criteria in the metallurgical industry, where aluminum powder is used as a heat source as well as a reducing agent. Such applications include production of ferro-alloys like ferro-columbium, pure metal, such as chromium and nonferrous allovs like boron-chromium. Other exothermic applications include hot topping compounds, stress relieving, exothermic welding, and powder lancing.

Powder compounds derived from aluminum powders also find a wide variety of uses in the chemical and plastics industries. These uses range from highly selective catalysts through powerful reducing agents to inert fillers. Aluminum alkyls, produced from powders, are a group of compounds used as feedstock in the production of biodegradable detergents and plasticizers. Aluminum chlorhydroxide and aluminum glycinate are widely used constituents in deodorant and antiperspirant formulations. As a filler for epoxy resins, aluminum powders can reduce shrinkage, increase thermal conductivity by an order of magnitude and reduce thermal expansion as well as reduce permeability and swelling attributable to water, oxygen, or other penetrants.

Powders flattened into tiny flakes by steel balls in a rotating mill provide the metallic pigments for paints and coatings. Leafing pigments, processed to rise to the surface of paint, reflect heat in roof coatings and can protect nonaluminum structures, such as bridges and tanks, from the weather. Nonleafing pigments, processed to remain suspended in paint or coating, provide the metallic finish for cars, trucks, and other items, such as inks and football helmets. The ability to tailor the pigment size and shape to achieve variations in appearance gives product developers an extra degree of freedom in design.

In the electronics area, aluminum nitride PM ceramics are excellent candidates for electronic packages and substrates, especially with multichip modules, because of thermal expansion and heat transfer characteristics. The use of improved thermal conductivity to yield a reduction in operating temperatures of a typical module by 10 °C (20 °F), will double the reliability of the circuit.

The most exciting application for aluminum powders is in the production of PM parts for structural and nonstructural applications in the transportation and commercial areas. These press and sinter products, blends of aluminum and elemental alloy powders, are pressed into intricate configurations and sintered to yield net or near-net shapes. There are two basic classes of commercial press and sinter aluminum alloys: 6061 (Al-0.25Cu-0.6Si-1Mg) and 2014 (Al-4.4Cu-0.8Si-0.5Mg). Alloy 6061 displays moderate strength (tensile strength is 237 MPa, or 34.5 ksi) with excellent corrosion resistance, while alloy 2014 develops high mechanical properties (tensile strength is 331 MPa, or 48 ksi) in both the as-sintered and heat-treated conditions.

Parts produced from these materials have the exceptional properties associated with aluminum: light weight, corrosion resistance, high thermal and electrical conductivity, good machinability, and excellent response to a variety of finishing processes. They also offer competitive raw material prices on a cost per unit volume basis.

However, the primary driver for the application of PM aluminum is the ability to produce complex net or near-net shape parts that need minimal or no machining. This capability makes PM parts cost-competitive with many castings, extrusion, forgings, and machinescrew products. In addition, the PM parts can be further processed by hot or cold forging to eliminate porosity and improve strength. Mechanical properties of such products compare favorably with those of conventional wrought alloys and are superior to those of typical press and sinter parts. The niche for PM forgings is in the strength/cost gap between castings and conventional forgings, and in applications such as connecting rods, gears, and pistons.

Newer aluminum powder products take advantage of rapid solidification technology to produce prealloyed PM materials with strength, toughness, fatigue, corrosion resistance, and elevated temperature performance not achievable with conventional wrought alloys. These powders can be blended with nonmetallic powders to produce PM-based metal-matrix composites (PM MMC) that have "next generation" properties in the areas of stiffness, fatigue, wear, and physical property control. Such new materials have been aimed at the aerospace market and have found uses in both new aircraft platform production and aging aircraft retrofit programs. The relatively limited applications coupled with high projected costs have resulted in reduced commercial interest by both producers and users. The technology, however, stands ready to solve the tough next generation product needs, and the ultimate progression of market use from aerospace to the commercial area could generate significant, high volume applications.

Aluminum powder products span an incredible range of uses from beautification to national defense and from the mundane to the highly sophisticated. Life without these products would not be the same, and they are the key to exciting new developments.

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# **Conventional Powder Metallurgy Aluminum Alloys**

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ALUMINUM ALLOYS have the distinct advantage over ferrous alloys of low density plus high thermal and electrical conductivity. By using such alloys in the powder metallurgy (PM) process of component manufacturing, large quantities of complex parts having these properties can be made every efficiently. PM aluminum components have been made successfully for many decades, but the first highvolume-production part was the camshaft bearing cap ("cam cap"), first shipped to General Motors Corporation (now "Company", in Detroit, MI) in 1991 (Ref 1). Similar cam caps (Fig. 1) have been developed for other GM engines and for other automotive companies (Ref 2-4). More than 200 million cam caps are estimated to have been supplied to the automotive industry since the launch of this program, using PM as the preferred manufacturing process for such components. Figure 2 shows one of the largest and more complex cam caps manufactured by the PM process and illustrates the intricate detail that can be incorporated by this process. This manufacturing methodology is a relatively efficient and economic process, with a demonstrated capability to produce high quantities of aluminum components with a reasonable degree of precision (Ref 4-6).

The low density of aluminum (2.70 versus 7.87 g/cm<sup>3</sup> for steel), is an asset in applications

in which lightweight materials can assist in the efforts to reduce vehicular mass. Along with aluminum, the alloys of magnesium and titanium are excellent candidate materials for aerospace and vehicular applications because they offer the promise of lightweight structural components (Ref 4, 6–9). Lower-weight vehicles help to achieve the highly desired goal of reducing fuel consumption.

Aluminum is the most abundant metal in the earth's crust and is highly reactive with other elements. Its reactivity with oxygen and the stability of the resultant oxide, combined with the high interfacial strength between the oxide and the substrate, is why aluminum exhibits such excellent corrosion resistance. Even with very low partial pressures of oxygen, nascent aluminum will form a thin, stable surface oxide film. Any water present in the atomizing gas or chamber will also react with that surface and influence the surface oxide composition (Ref 10, 11). The aluminum oxide (alumina) barrier formed on the surface creates an effective shield to harmful fluids.

The relatively high thermal conductivity of aluminum makes the metal highly suitable for thermal management applications such as



Fig. 1 Examples of some small aluminum camshaft bearing caps produced by powder metallurgy



Fig. 2 PM aluminum camshaft bearing cap. 2006 Grand Prize award winner in the automotive category. Courtesy of Metal Powder Products, GM, and MPIF. Source: Ref 5

computer and amplifier heat sinks. The high electrical conductivity of the metal is the reason it is used extensively in electrical transmission cables. If aluminum components are used to terminate electrical wiring (i.e. connectors), care must be taken because an oxide film can form at the interface over a period of time. This can lead to an increase in electrical resistance at that joint and may result in excessive heating at the interface.

This technology is highly suitable for processing aluminum because the metal is so soft and ductile that it is possible to achieve reasonably high densities in the compacting step with lower compaction pressures than those used for ferrous powders. The sintering of aluminum presents certain difficulties because of the stability of its oxide. The normal practice of using hydrogen is not commercially viable since very dry atmospheres would be required to reduce the surface oxide to metal.

The primary objective of this article is to describe the application of the technology to the manufacture of aluminum components and highlight some of the current R&D work that is aimed at improving the structural properties of this alloy system. The vast majority of PM aluminum parts made to date have been produced with repressing (sizing) as the final operation of the PM process, although additional operations can be incorporated, as discussed below. In the PM industry, it is customary to refer to any process conducted after sintering as a "secondary operation," and such operations may be required for specific components and applications. Further examples of such secondary operations are anodizing, impregnation, and plating.

#### **Powder Production**

Several methods are available to produce powders; the one most widely used for compacting grade powders is air atomization of the molten metal. Pure aluminum or the alloy is poured from the furnace to a tundish, and the molten metal is

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usually fed by gravity through an orifice and atomized by high-pressure jets of air (Ref 12, 13). The resulting powder is collected and, if necessary, air-classified. The contents of the containers may be remixed to create specific particle size distributions. Since the powder production process is all done in air, there is a fine coating of aluminum oxide (alumina) on the particle surface. Further details and discussion of additional aluminum powder production methods are described in the article "Production of Aluminum and Aluminum Alloy Powder" in this Volume.

The powder form of aluminum should be handled with care because of the high specific surface area and the high affinity of the metal for oxygen. This pyrophoric nature of aluminum powder means that precautions should be taken to avoid generation of fine dust clouds, as well as to avoid ignition sources such as electrical sparks and electrostatic charges (Ref 14-15). National Fire Prevention Association (NFPA) Standard 654 provides guidance on the prevention of fire and dust explosions (Ref 16). This includes restrictions on cigarette smoking, open flames, sparks, and hot surfaces in closed areas that might generate or collect dust. As long as sensible precautions, such as grounding of equipment, are taken, aluminum powder can be handled in a safe manner, both in the powder production facilities and in the part-making plants, up to the compacting stage of the PM process. The conventional PM process requires the addition of a lubricant to the aluminum powder; this lubricant tends to provide a coating of organic material and makes aluminum powders stick together, making them less susceptible to forming dust clouds. Once aluminum has been compacted into a green component, the flammability risk is decreased, since the compact containing metal and lubricant is not likely to generate fine metal dust.

#### Compaction

Several molding-grade powders are available both in elemental and alloyed form. Metal powders are usually admixed with a lubricant to enable ejection of the green part once it has been die-compacted. Ethylene bis(stearamide) (EBS) wax is the preferred lubricant in the industry because the degradation products are environmentally friendly (primarily water vapor, CO, and  $CO_2$ ). Experience has shown that quite high levels of lubricant (1.5% by weight) are needed in order to avoid compaction defects. In a few instances, some parts producers prefer to use die-wall lubrication instead.

Pure aluminum metal powder may be combined with other additives such as copper, silicon, magnesium, and zinc to form specific alloy compositions. Examples of some common aluminum PM alloys are given in Table 1.

The table generally follows the four-digit designation method for wrought aluminum alloys, in which the first digit specifies the major alloying element that is added to the aluminum and therefore defines that alloy series:

- 1yyy: pure aluminum
- 2yyy: copper added
- 3yyy: manganese added
- 4yyy: silicon added
- 5yyy: magnesium added
- 6yyy: both magnesium and silicon added
- 7yyy: zinc added
- 8yyy: other elements added

For the pure aluminum series only, the last two digits define the minimum aluminum percentage above zero, i.e. the purity of the material. For example, the alloy designation 1050 signifies that the aluminum is at a minimum purity level of 99.50%. For all the other alloy systems, the last two digits denote an arbitrarily assigned alloy number, not related to composition. The second digit, if different from 0, denotes a modification of a specific alloy; for example, though alloys 3003, 3004, and 3005 are different from each other, alloy 5352 is a derivation or modification of alloy 5252 and alloy 5052. The Registered International Designations and the official registered compositions for wrought and cast aluminum alloys are maintained in the United States by the Aluminum Association.

In PM, the additional variable is density. which is controlled by the compaction pressures and the sintering protocols used. Aluminum powders are highly compressible, or malleable, compared to iron and copper powders (Fig. 3), so that a higher green density is achievable in aluminum for a given applied compaction pressure. For example, by using a typical compaction pressure of 207 MPa (15 tsi) a density equivalent to 90% of the full or pore-free density, termed *relative density*, can be achieved in aluminum. The corresponding values for iron and copper at the same pressure are less than 70% of their respective pore-free densities. In fact, to achieve the same 90% relative density in iron, a compaction pressure of 620 MPa (45 tsi) is needed. The practical implication of this is that larger PM parts can be made of aluminum on a given press size than of iron or copper. Another benefit is that the aluminum powder is able to flow during cold compaction and form intricate and precise details; examples of this are the grooves in the cam caps shown in Fig. 1 and 2.

Commercially available tool steels or carbides can be used as materials for tooling when compacting aluminum powders into green compacts. Slightly higher tooling wear than that for iron might be experienced with aluminum powders because of the thin abrasive aluminum oxide film that exists on all aluminum powder surfaces.

In addition to forming the green part shape, the compacting step serves to break up the surface oxide and create metal-to-metal bonds. This is quite important for aluminum powders because of the high stability of the oxide, which has to be broken down before acceptable sintering can occur. The metal-to-metal bonds and the interlocking of particles during compaction result in values of green strength of  $\sim 10$  MPa (1,500 psi). Green parts can be handled readily in an industrial environment as long as reasonable care is taken during material transfer between the compaction and sintering stages.

 Table 1
 Nominal compositions for common PM aluminum alloys employed in commercial manufacturing operations

Designation			Composition, wt%						
ASTM	Alternate	Al	Cu	Mg	Si	Zn	Others(a)		
A-1050		bal							
A-2014	AMPAL AMB2712, ECKA Alumix 123, (Obsolete ALCOA 201AB)	bal	3.8-5.0	0.2–1.2	0.5–1.2		1.5		
A-2214	(Obsolete ALCOA 202AB)	bal	3.8-4.2				1.5		
A-2314	(Obsolete MD-22)	bal	1.8 - 2.2	0.8 - 1.2	0.3		1.5		
A-4032	AMPAL AMB40XX	bal	0.8 - 3.5	0.4-1.20	6-15		1.5		
	ECKA Alumix 231	bal	2.4 - 2.8	0.5-0.8	14-16		1.5		
A-5106		bal	0.1-0.5	0.8 - 1.2	0.2-0.6		1.5		
A-6061	AMPAL AMB6711, ECKA Alumix 321 (Obsolete ALCOA 601AB; MD-69)	bal	0-0.4	0.8-1.2	0.4–0.9		1.5		
A-6002	(Obsolete ALCOA 602AB)	bal		0.4-0.8	0.2-0.6		1.5		
A-7075	AMPAL AMB777X	bal	0.7 - 1.7	2.0-3.0	0-0.6	6.0-8.0	1.5		
	ECKA Alumix 431	bal	1.5-2.0	2.2-2.8		5.6-6.4	1.5		

(a) Value for the combined concentrations of admixed lubricant, typical impurities (oxygen, nitrogen, iron, etc.), and any deliberate trace additions included for enhanced sintering response and/or mechanical performance (tin, zirconium, chromium, etc.)



Fig. 3 Compressibility curves for aluminum, copper, and ferrous-base powders, demonstrating the highly malleable response of aluminum powders Sintering is a critically important step in the PM process since this is the stage in which the usable properties of the metal or alloy system are developed. This step is even more important for aluminum alloy systems because of the high stability of the metal oxide film that encases the individual powder particles.

Once the first stage of lubricant removal is complete, the next significant stage is the bonding of metal particles. In order for metal-to-metal bonds to develop, the particle surface oxides must be reduced by some means. The highly stable oxide on the surface of aluminum powders is a challenge for conventional PM sintering practices, wherein hydrogen and low dew points are often leveraged to successfully remove this feature. From a thermodynamic perspective, reduction of aluminum oxide at 600 °C (1110 °F) can occur only if the furnace atmospheric dew point is at -140 °C (-285 °F), which is equivalent to an oxygen particle pressure of less than  $10^{-45}$ kPa ( $10^{-50}$  atm.) (Ref 17, 18).

The aforementioned conditions are impractically low values for an industrial environment. Hence, surface activation techniques are employed (Ref 19-28) to enable the development of metalto-metal bonds. Many of the commercially available PM aluminum alloys contain small amounts of magnesium for this purpose. This element serves to partially reduce the surface film of alumina by forming the spinel MgAl<sub>2</sub>O<sub>4</sub>. As little as 0.1% admixed magnesium is needed to initiate this reaction in the solid state (Ref 19, 24, 27). Other elements in small quantities can also serve to activate compact sintering. For instance, trace additions of tin, lead, indium, bismuth, and antimony intensify sintering in the Al-Cu-Mg alloy system (Ref 19, 20).

Densification and chemical homogenization are also important. These transitions within the compact are driven by the presence of a liquid phase that can be of a transient or persistent character depending on the alloving additions involved and the nature of the raw powder blend. Typically, aluminum PM alloys are designed to exhibit a persistent liquid phase as this serves to improve process robustness. Termed liquid phase sintering (LPS), this process offers many advantages, including accelerated mass transport and capillary pull among the solid powder particles, effectively behaving as an external pressure to densify the sintering compact (Ref 28). Numerous alloying elements can be employed for this purpose, including copper (Ref 23), silicon (Ref 26), magnesium (Ref 27), and zinc (Ref 29). Lumley and Schaffer (Ref 21) have shown that both the amount and the particle size of the additives used for liquid formation must be controlled carefully in order to achieve acceptable levels of dimensional control. Consolidation during sintering is an important objective to improve mechanical properties, but can be counterproductive if this results in excessive distortion, leading to poor dimensional control.

Clearly, the sintering process for PM aluminum is quite complex, and component dimensions vary throughout this thermal cycle. For instance, the compacted part may grow first as the additives are dissolved into the aluminum powder and liquid formation causes particle separation. This is then commonly followed by appreciable shrinkage as the liquid phase invokes densification mechanisms such as particle rearrangement (Ref 19, 27, 30, 31).

Aluminum PM green parts can be sintered in protective furnace atmospheres such as pure nitrogen, nitrogen plus hydrogen, vacuum, or dissociated ammonia. However, studies have shown that nitrogen is the best atmosphere for sintering aluminum; Schaffer and Hall (Ref 32) believe that at low green part densities, nitrogen can play an even greater role in the reduction of  $Al_2O_3$  than magnesium because of the formation of aluminum nitride (AlN). This nitride formation is the reason why sintering in nitrogen is even more effective than in an inert gas such as argon or vacuum (Ref 32-35). Hydrogen appears to have a negative effect on the sintering of aluminum alloys, although the exact mechanism is unknown (Ref 33-35). One possibility is that hydrogen hinders the formation of AlN by reacting with oxygen and forming water vapor. The oxide of magnesium is even more stable than that of aluminum (Ref 17, 18), enabling magnesium to act as a getter that reduces the oxygen level in the sintering atmosphere to such a low level that AlN can form (Ref 25, 32).

The specific temperature and thermal profile to be used for sintering are dependent on the alloy composition and the additives used to aid bonding and consolidation, but typical temperatures are in the range of 570 to 630  $^{\circ}$ C (1070 to 1170  $^{\circ}$ F). Time at sintering temperature typically ranges from 10 to 30 minutes (Ref 4, 30, 31).

#### Repressing

The dimensional change in the sintering furnace for PM aluminum is usually significantly higher than that for most ferrous-base systems. Therefore, repressing is a routine postsinter step in the PM process to manufacture aluminum parts by this methodology (Ref 20). The operation is called *sizing* if the main goal for repressing the sintered part is to improve component dimensional accuracy; if the main goal is to create additional features on the part, it is called *coining*. For example, counterbores or small details can be incorporated in the tooling punch faces and coined in on the part faces, as long as their depth is no more than 10% of the part length in the pressing direction.

If extensive deformation takes place during the repressing stage and increased densification is a primary goal, a heat treatment step may be needed to soften the aluminum alloy. Additional consolidation may be achieved by resintering, depending on the specific alloy composition and the time and temperature used. The soft and ductile nature of PM aluminum alloys means that cold or hot forging of the material is possible and may be required for high-performance applications.

The predominant aluminum alloy processed by conventional powder metallurgy, Al-4%Cu-1% Si-0.5%Mg, is fairly soft and ductile in the assintered condition and begins to harden (age) fairly quickly at room temperature, attaining essentially full hardness in a day or so. For this reason, any repressing operation should be performed in a matter of hours following the sintering step.

#### Heat Treatment

As shown in Table 1, compositions of the commercial aluminum PM materials can be quite complex. The types and concentrations of alloying additions are carefully chosen as they have a decisive effect on the compaction and sintering stages of the PM process. For example, magnesium plays a pivotal role in disrupting the oxide film on aluminum particles, and copper is important in liquid phase formation, as discussed earlier. Many of these elements also impart heat-treatable characteristics to the finished PM products. Heat treatment of aluminum products can be completed through numerous approaches, including:

- T1: as-sintered and naturally aged (at room temperature)
- T2: cold worked and naturally aged
- T3: solution treated, cold worked, and naturally aged
- T4: solution treated and naturally aged
- T5: artificially aged only (at a slightly elevated temperature)
- T6: solution treated and artificially aged
- T7: solution treated and stabilized (overaged)
- T8: solution treated, cold worked, and artificially aged

Of these various options, T2 is the most popular for the vast majority of aluminum parts being currently manufactured by PM. The T1 and T2 tempers are the most cost-effective heat treatments, and they offer attractive mechanical properties in the sintered product. Favorable economics stem from the fact that solutionization is included as an integral element of the sintering cycle, avoiding the need for a separate secondary reheating operation with the associated increase in capital costs. At the peak sintering temperature, aluminum PM compacts are in a semisolid condition. The solid fraction is dominant and includes a significant proportion of the alloying elements dissolved in solid solution. After the prescribed time at the sintering temperature, the compacts are gas-cooled to ambient temperature, in a water-jacketed section of the furnace, while under the protective atmosphere. These cooling rates are sufficient to preserve the  $\alpha$ -aluminum grains of the material in a metastable supersaturated condition upon exiting the furnace (Ref 36). This permits conventional precipitation sequences to transpire during subsequent aging at room temperature, resulting in an increase in apparent hardness as well as in other properties such as tensile yield strength. An example of the postsinter natural age-hardening response typical of A-2014 is shown in Fig. 4. Here, a hardness increase of  $\sim$ 15% transpires within two days after sintering. All conventional aluminum PM alloys behave in a similar manner, although the extent and kinetics of the hardness transition varies with alloy chemistry.

When even greater mechanical performance is required, aluminum PM parts can be heat treated to the T4 or T6 temper. These processes are typically applied to the sintered product after it has been cooled to ambient temperature and sized to refine dimensional tolerances. Each of these tempers includes a much longer solutionization time than that in the T1 temper. This promotes increased diffusion of alloying elements into solid solution and enhances microstructural homogeneity. This is followed by a rapid water quench and natural (T4) or artificial (T6) aging to the condition of a stable peak hardness. Artificial aging curves that illustrate the typical response of conventional aluminum PM alloys are shown in Fig. 5.

Table 2 shows the mechanical properties of some of the more common PM aluminum alloys used in current commercial practice, and displays the effect of the described thermal treatments.

Investigations of the heat treatment response of traditional wrought and cast aluminum alloys have revealed significant insight on the various precipitation sequences that can transpire. The dominant sequence is governed by the alloy chemistry and the relative concentrations of the different elements present. For instance, in Al-Cu-Mg alloys with a high copper/magnesium ratio, strengthening is principally achieved through the formation of  $\theta$  (Al<sub>2</sub>Cu) phases (Ref 40). However, when the copper and magnesium concentrations are more evenly balanced, S (Al<sub>2</sub>CuMg) phases prevail as the leading strengthening feature (Ref 41). Likewise,  $\eta$  (MgZn<sub>2</sub>) and  $\beta$  (Mg<sub>2</sub>Si) precipitates are generally the key phases observed in Al-Zn-Mg (Ref 42) and Al-Mg-Si alloys respectively (Ref 43).



Fig. 4 Aging response of A-2014. Apparent hardness as a function of time after sintering. Source: Ref 37

A number of studies have also assessed the precipitates in conventional aluminum PM alloys. For instance, transmission electron microscopy (TEM) work on A-2014 has confirmed that sintered products in the T6 temper

condition are principally strengthened by  $\theta'$  precipitates, whereas those in a T4 condition contain high concentrations of Guinier-Preston (GP) zones (Ref 44). Such observations are in direct agreement with studies on the counterpart



Fig. 5 Examples of artificial aging curves developed for conventional PM materials. (a) A-2014. (b) A-4032. (c) A-7075. Source: Ref 38

	Compaction	Green	Der	nsity		Vield			
Alloy	pressure, MPa (ksi)	strength, MPa (ksi)	Green % (g/cm <sup>3</sup> )	Sintered % (g/cm <sup>3</sup> )	Temper	strength, MPa (ksi)	UTS, MPa (ksi)	Elongation, %	Hardness
A-2014	110 (8)	4.2 (0.6)	85 (2.36)	91.0 (2.53)	T1	145 (21)	169 (25)	2	60 HRE
					T4	179 (26)	210 (31)	3	70 HRE
					T6	248 (36)	248 (36)	<1	80 HRE
	180 (13)	8.3 (1.2)	90 (2.50)	93.0 (2.58)	T1	170 (25)	201 (29)	3	70 HRE
					T4	205 (30)	245 (36)	3	75 HRE
					T6	322 (47)	323 (47)	<1	85 HRE
	413 (30)	13.8 (2)	95 (2.64)	97.0 (2.70)	T1	181 (26)	209 (30)	3	70 HRE
					T4	214 (31)	262 (38)	5	80 HRE
					T6	327 (48)	332 (48)	2	90 HRE
A-2214	180 (13)	5.4 (0.8)	90 (2.49)	92.4 (2.56)	T1	75 (11)	160 (23)	10	55 HRH
					T4	119 (17)	194 (28)	8	70 HRH
					T6	147 (21)	227 (33)	7	45 HRE
A-4032	600 (44)	13 (1.9)	91.8 (2.46)	98.5 (2.64)	T1	206 (30)	206 (30)	1	40 HRB
					T6	330 (48)	339 (49)	<1	83 HRB
A-6061	96 (7)	3.1 (0.5)	85 (2.29)	91.1 (2.45)	T1	48 (7)	110 (16)	6	55 HRH
					T4	96 (14)	141 (20)	5	80 HRH
					T6	176 (26)	183 (27)	1	70 HRE
	165 (12)	6.6 (1.0)	90 (2.42)	93.7 (2.52)	T1	88 (13)	139 (20)	5	60 HRH
					T4	114 (17)	172 (25)	5	80 HRH
					T6	221 (32)	232 (34)	2	75 HRE
	345 (25)	10.4 (1.5)	95 (2.55)	96.0 (2.58)	T1	94 (14)	145 (21)	6	65 HRH
					T4	117 (17)	176 (26)	6	85 HRH
					T6	230 (33)	238 (34)	2	80 HRE
A-6002	165 (12)	6.6 (1.0)	90 (2.42)	93 (2.55)	T1	59 (8)	121 (17)	9	55 HRH
					T4	62 (9)	121 (17)	7	65 HRH
					T6	169 (24)	179 (26)	2	55 HRE
	345 (25)	10.4 (1.5)	95 (2.55)	96 (2.58)	T1	62 (9)	131 (19)	9	55 HRH
					T4	65 (9)	134 (19)	10	70 HRH
					T6	172 (25)	186 (27)	3	65 HRE
A-7075	200 (15)	4.5 (0.7)	80.6 (2.27)	97.7 (2.75)	T1	221 (32)	241 (35)	2	55 HRB
					T6	463 (67)	499 (72)	1	87 HRB
	400 (29)	7.0 (1.0)	88.6 (2.49)	97.7 (2.75)	T1	279 (40)	323 (47)	1	67 HRB
					T6	473 (69)	497 (72)	1	87 HRB
Adapted fro	om Ref 39								

## Table 2 Typical mechanical properties for common PM aluminum alloys employed incommercial manufacturing operations

wrought alloy A-2014-T6 (Ref 45), Differential scanning calorimetry (DSC) studies on the Al-Zn system have confirmed that commercial PM A-7075-T6 is principally strengthened by GP zones formed during the precursory step of the n precipitation sequence (Ref 46). This was also a direct replication of findings on the corresponding wrought alloy heat treated to the same state (A-7075-T6). In both instances, the presence of residual porosity and a sintering additive (i.e. trace additions of tin) were found to have no influence on the precipitates formed. Such studies confirm that the heat treatment behavior of aluminum PM products is well aligned with that of traditional wrought/cast materials of similar bulk chemistries.

#### **Emerging Aluminum PM Materials**

Though much progress has been made over the years in commercialization of the PM aluminum technology in support of automotive cam caps. emerging opportunities require a combination of properties that may not be accommodated with the existing suite of conventional systems. Hence, the development of new aluminum PM materials that expand the collective portfolio of mechanical and physical properties commercially available is of significant industrial interest. A series of emerging alloys have been devised through academic and industrial research dedicated to this initiative. All of the systems discussed below have matured to a point at which industrial viability has been firmly established through the recent launch of new high-volume components or the successful execution of pilot scale production runs.

Enhanced Tensile Ductility. Conventional aluminum PM alloys typically exhibit tensile ductility in the range of 0 to 5% (Table 2). Some special materials offer higher levels but also have relatively low yield strengths, rendering them unsuitable for structural applications. More desirable aluminum PM materials are those that offer improved ductility without compromising yield strength. A number of the emerging materials that exhibit such attributes are listed in Table 3. Two of these examples are modified versions of conventional A-2014-T1 and A-6061-T1 (Ref 47). Both exhibit significant increases in ductility without compromising yield strength or ultimate tensile strength (UTS). The third alloy is an entirely new formulation that employs a relatively low copper/magnesium ratio as a means of attaining an improved balance of properties (Ref 48).

**Responsiveness to Sizing Operations.** The fabrication of engineered components through aluminum PM technology is dominated by a three stage process: die compaction, liquid phase sintering, and sizing. While a growing number of research programs are now focused on aluminum PM, the sizing stage of the process has been afforded minimal attention. Sizing is an important step in the overall production sequence, as it permits achievement of tight dimensional

tolerances, increased density, and improved mechanical properties in the finished product. Metallurgically, sizing is a cold forming operation applied to sintered compacts. It is typically carried out immediately after sintering, before the postsinter natural age hardening and the accompanying reduction in tensile ductility (Ref 3, 37).

Similar procedures are employed with select wrought aluminum alloys. Here, the alloy is fully solutionized, water quenched, cold worked, and naturally aged. This T3 tempering is akin to the T2 process followed in aluminum PM wherein components are sintered, gas cooled, sized, and then naturally aged. The T3 process is applied only to those wrought alloys that are strengthened by precipitates that preferentially nucleate on dislocations. Formed during the cold working stage, the dislocations promote a refined spatial distribution of any phases that subsequently precipitate from supersaturated solid solution during natural aging. The net effect is a considerable gain in tensile and fatigue properties over those produced by processes without the coldwork stage (Ref 49). The wrought Al-Cu-Mg alloys responsive to this approach are almost all strengthened by S precipitates (Al<sub>2</sub>CuMg); those that rely solely on the precipitation of other phases, such as  $\theta$  (CuAl<sub>2</sub>), do not display this response. The relative amounts of precipitates developed from the  $\theta$  and S sequences are principally dictated by thermodynamic factors and bulk alloy chemistry. The former are the dominant strengthening agents when a high copper/magnesium ratio exists (Ref 50), whereas the latter prevail in those with an appreciably lower ratio (Ref 51). This concept is equally applicable to conventional PM aluminum Al-Cu-Mg alloys. For instance, A-2014 has a relatively high copper/magnesium ratio of 7.5:1 and is known to favor the precipitation of  $\theta$  phases with an effective absence of those from the S family (Al<sub>2</sub>CuMg) (Ref 44).

Given that cold work induced by sizing is intrinsic to high volume aluminum PM processing, efforts have been made to design new Al-Cu-Mg alloys such that S precipitates are promoted. One key example is an emerging PM alloy with a copper/magnesium ratio of 3.0 (Al-4.5Cu-1.5Mg) (Ref 52). This alloy is highly responsive to die compaction and exhibits excellent densification during standard sintering cycles in laboratory and industrial settings (Ref 52, 53). A bright-field (BF) transmission electron microscopy (TEM) image representing a typical sintered microstructure of the alloy is shown in Fig. 6. It is apparent that a homogenous distribution of lath shaped particles is the dominant secondary phase. These particles appear from their size and orientation to be S' precipitates. This conclusion is further substantiated by a selected-area diffraction pattern (SADP) of the lath-shaped particles, which reveals the presence of "crosses" about the  $[110]_{\alpha}$  positions, a feature characteristic of the S' phase (Ref 54, 55).

Typical mechanical properties of the Al-4.5Cu-1.5Mg alloy are shown in Table 4. Comparative data from test bars of A-2014

 Table 3
 Overview of emerging aluminum PM alloys that demonstrate enhanced tensile ductility

All data derived from as-sintered dog bone specimens in the T1 condition

	Sintered density				
Alloy	g/cm <sup>3</sup>	%	Yield, MPa (ksi)	UTS, MPa (ksi)	Elongation, %
A-2014-T1 (modified)	2.74	98.6	200 (29)	270 (39)	5
A-6061-T1 (modified)	2.69	99.9	100 (15)	170 (25)	15
A-2618-T1	2.71	99.7	207 (30)	322 (47)	10
B 6 45 49					

Source: Ref 47, 48



Fig. 6 TEM results acquired from PM Al-4.5Cu-1.5Mg. (a) BF micrograph recorded in the  $[001]_{\alpha}$  orientation showing lath-shaped S precipitates along  $[100]_{\alpha}$  and  $[010]_{\alpha}$  directions. (b) Corresponding SADP showing the "cross"-like intensity distribution characteristic of the 12 orientational variants of the S' phase