Powder Technology 226 (2012) 225–230

Contents lists available at SciVerse ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Surface porosity investigation of aluminum–silicon PM alloys

G. Steedman, D.P. Bishop, W.F. Caley, G.J. Kipouros *

Materials Engineering Program, Department of Process Engineering and Applied Science, Dalhousie University, 1459 Oxford Street, Halifax, NS, Canada B3H 4R2

ARTICLE INFO

Article history:
Received 15 June 2011
Received in revised form 8 January 2012
Accepted 26 April 2012
Available online 5 May 2012

Keywords:
Surface porosity
Open porosity
Powder metallurgy
Aluminum–silicon alloys
Pycnometry
Surface engineering

ABSTRACT

An alternative method for measuring the surface porosity of pressed and sintered PM alloys was investigated. Materials used for this research consisted of a commercially available alloy, Alumix 231, and a proprietary alloy, DAL 6-Si PM alloys. The densities of the samples of the two alloys were analyzed using helium-pycnometry and oil-impregnation. In both cases, the measured density by helium-pycnometry was found to be 1–2% higher than that determined by the oil impregnation method and closer to the theoretical density. This is believed to be due to the superior ability of helium to penetrate the microstructure of these alloys. In addition, the volume of open (surface) porosity was calculated for both alloys. The results showed significant differences in the volume of open and closed porosity measured by the two techniques. Helium-pycnometry gives higher open porosity values and lower closed porosity compared to oil impregnation method.

1. Introduction

The use of powder metallurgy (PM) alloys is continually expanding. In particular, aluminum PM alloys are being used in several automotive applications in place of more traditional ferrous alloys. Their use is part of a trend toward materials that can reduce the weight of the vehicle. PM is able to produce high strength aluminum alloys that generally have mechanical properties comparable to structural steels [1].

Interest in aluminum powder metallurgy was renewed in the 1990's when camshaft bearing caps were first produced for General Motors and DaimlerChrysler. Fabrication of these products annually exceeding 10^7 units per year for a single engine program is a routine. With the success of these initial products the interest is shifting into manufacturing of pump gears, thrust plates, connecting rods, variable cam phasers, and retainer plates. Each new application is accompanied by a unique portfolio of mechanical demands for attributes such as tensile/compressive properties, elevated temperature strength/stability, fatigue resistance, and tribological performance the latter two been very sensitive to surface finishing and surface porosity. In addition corrosion resistance is always a requirement for applications under the automobile hood. Among the advantages of the aluminum powder metallurgy in the "press and sinter" mode is the manufacture of mechanical components with significant geometrical complexity and tight dimensional tolerances (i.e. ±20 μm) [2,3].

While there are several factors limiting further use of aluminum PM alloys, the corrosion performance is one of the most significant. It should be noted that there is no reliable method to assess the corrosion behavior of PM alloys. Many investigators have attempted to do so [4], but the conclusions drawn of the corrosion performance have been inconsistent and unreliable. This is due to uncertainty about the exposed surface area of the sample and the corrosion mechanisms that are occurring on the surface of these alloys due to morphology of the surface. The surface morphology of PM alloys is formed during the consolidation that occurs between the powder particles during compaction and sintering and results in an uneven and open surface with possible interconnected porosity. When compared to wrought alloys of similar composition, the PM alloys appear to be inferior in corrosion performance in several key areas. However, no comparison has taken into account the actual surface area and the surface morphology of the PM alloys and both are compared on the assumption of equal apparent surface area. This is significant because it is estimated that the actual surface area of PM alloys may be several orders of magnitude larger than the surface area of the wrought alloys [5] of equivalent apparent surface area. This creates a much larger exposed area for the PM alloys in a corrosive environment. The surface morphology of these alloys may also allow crevice corrosion to occur. In addition, the relative pH between the surface of the alloy and of an exposed pore is too different, causing the pore to act as an anode relative to the surface [6].

In order to accurately determine the corrosion performance of PM alloys, the surface area and morphology of these alloys must be investigated. In particular the actual surface area of PM alloys must be quantified, as well as the size and distribution of the surface porosity. In addition, the existence of certain corrosion mechanisms such as crevice corrosion and pitting must be investigated or predicted by image modeling. The first step of this research is to accurately quantify the open porosity that exists in the PM alloys.

* Corresponding author.
E-mail address: georges.kipouros@dal.ca (G.J. Kipouros).

0032-5910/$ – see front matter
doi:10.1016/j.powtec.2012.04.049

1.1. Helium pycnometry

Gas-pycnometry is a technique used for measuring the volume of a known weight sample of loose powders. Using a fluid such as helium, this technique uses pressure equalization to calculate volume. By using helium, the smallest pores of a material can be occupied due to helium's small atomic size, close to one Angstrom (10^{-10} m) in size [7]. There is also negligible tendency for adsorption of helium in the pores of the material at room temperature. The calculated density from helium pycnometry can be combined with a bulk density measurement to determine open and closed porosity of a sample material.

The test apparatus for helium pycnometry generally consists of two cells of known volume connected in series with a pressure monitoring device between them. The first chamber is the sample cell where the previously dried powder sample is placed, and the second is a reference cell. The cells can be isolated from each other and the external environment with a series of valves. The empty sample cell volume is first calculated for calibration. The sample cell is isolated with the pressure sensor set to 0, and then pressurized. The pressure is recorded once it has stabilized with the supply closed. The connecting valve between the sample and reference cells is then opened allowing the pressure to equalize. This second pressure reading is recorded. Using Boyle's Law, the volume of the sample cell can be calculated assuming ideal gas behavior. The assumption of ideal behavior is accepted as valid at the temperatures and pressures used for this measurement [8]. Fig. 1 illustrates a basic schematic for a pycnometer.

Once the volume of the empty sample cell has been calibrated, a dried, weighed sample can be placed inside the sample cell. The same procedure is repeated in order to calculate the new volume of the sample cell. With the volume of the sample cell calculated containing a sample, subtracting the volume of the empty sample cell from the occupied volume will yield the volume of the sample. The volume calculated by this technique is commonly described as the "true" volume, meaning that all open surface features have been occupied by the fluid. Pycnometry has been used primarily for highly porous materials, such as powders and sands. However, recently, attempts have been made to apply this technique to PM ferrous alloys [9].

1.2. Oil impregnation

Oil impregnation is the standard test method for determining the sintered bulk and true density of PM samples (ASTM B963, MPIF Standard 42). For this technique, the pre-weighed samples (M_{air}) are immersed in oil in a vacuum chamber. A vacuum is pulled and any air pockets within the microstructure are replaced with oil. The sample is then left in the vacuum chamber for 30 min to ensure that all air pockets have been evacuated. The oil-impregnated sample is then removed from the vacuum chamber, weighed (M_{oil}), and then immersed in distilled water to determine the oil-impregnated mass ($M_{oil+water}$). Using Archimedes' principle, the bulk density of material is calculated by applying the appropriate equation.

The density determined by both helium-pycnometry and oil impregnation is often cited as free of open porosity, meaning all surface features are infiltrated by the measuring fluid. However, any measured differences between the two methods may indicate a deficiency in either with measuring porosity. Therefore, for this research, the density measured by each technique is labeled individually: $\rho_{oil\text{-}impregnation}$, and $\rho_{pycnometry}$.

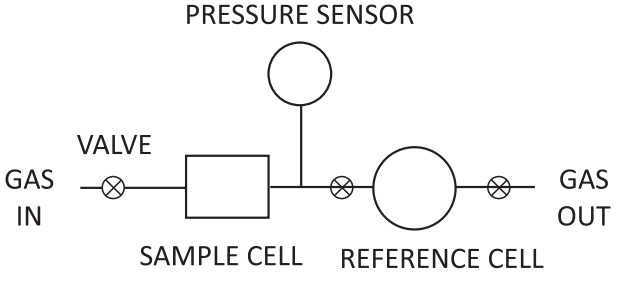

Fig. 1. Pycnometer schematic.

2. Materials

Two aluminum–silicon PM alloys were used. The first, Alumix 231 is a commercially available PM alloy that was supplied by Ecka Granules Inc and its mechanical properties, in high density formulation, matches those of its wrought counterpart AA4032 in yield strength, ultimate tensile strength (UTS) and hardness. This powder blend contained a mixture of nearly a pure aluminum powder, and a pre-alloyed powder containing aluminum and all the other elemental additions [10]. Prior to compaction, the powder was re-blended in a Turbula mixer-shaker to ensure that the mixture was completely homogenous prior to compaction. The second PM alloy was a proprietary alloy developed in our laboratory at Dalhousie University, DAL 6-Si and it represents the low silicon promising equivalent. The powder mixture for this alloy had been prepared previously, and only required re-blending in the Turbula prior to compaction. The powders used in the DAL 6-Si alloy were: elemental aluminum, copper, and tin, and Al–Si and Al–Mg master alloy powders.

Compositionally, the two alloys differ primarily in the silicon content, as this is the main alloying element in both alloys. Other differences were in copper content and the addition of tin in the DAL 6-Si alloy. Table 1 shows the composition of the two alloys.

Additionally, both blends contained 1.5w/o Licowax to reduce die wear in compaction. The compaction and sintering of both alloys was performed under optimal conditions. The sintering and compaction conditions for Alumix 231 were modified from the conditions recommended by Ecka Granules Inc [11], while the optimal sintering conditions for DAL 6-Si had been determined previously [12].

3. Experimental procedure

3.1. Compaction

The compaction of the two PM alloys was performed using a SATEC model 5594–200 HVL 1 MN load frame. Both alloys were pressed to a pressure of 600 MPa. This pressure had been determined earlier by constructing the corresponding compaction curves to be the optimal pressure for compaction of both alloys. Transverse rupture strength bars (TRS) were produced using a uni-axial die [13]. These bars required approximately 10 g of powder. Following the completion of the compaction, all samples were weighed and the thickness (OAL) was measured.

3.2. Sintering

Sintering of both alloys was performed using a Lindburg Blue 3-Zone Tube Furnace. The progress of the sintering process was monitored using a K-type thermocouple. Once the sample tray had been moved into position in the furnace, a vacuum was created within the tube to ~10^{-3} Torr. High-purity nitrogen was introduced into the tube and then evacuated in order to remove any oxygen trapped within the

Table 1
As-mixed powder composition (w/o) excluding lubricant.

Powder Alloy	Al	Si	Cu	Mg	Sn
Alumix 231	82.9	14	2.5	0.6	–
DAL 6-Si	88.8	6	4.5	0.5	0.2