TEMPERATURE PROGRAMMED DESORPTION STUDIES OF OUTGASSING OF ALUMINIUM POWDER

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The mechanical properties of powder metallurgy (P.M.) aluminium alloys may be influenced considerably by the hydrogen content. To obtain high-quality alloys, it is necessary to optimize the degassing of the powder before sintering. Such a process involves an understanding of the mechanism of the desorption of H2 from the surface of the powder.

By means of the temperature programmed desorption technique, hydrogen evolution has been studied on an X7xxx (Al, Zn, Mg, Cu) P.M. alloy. The results show a large influence of the atomization procedure (with air or with helium). Hydrogen formation results from water decomposition at the contact of the alloying metals, but two different mechanisms may occur.

— Water is chemisorbed on the sample and the decomposition process is conditioned by the break-up of the oxide layer, followed by the evaporation of zinc and magnesium.
— As a function of the temperature, water is liberated in the network of the oxide by a transformation of the aluminium hydroxides and may react with metal at the metal-oxide interface.

It is possible to improve the mechanical properties of aluminium alloys through powder metallurgy (P.M.). The rapid solidification of the atomized droplets allows an increase in the solubility of the alloying elements and refinement of the microstructure. However, the ductility of P.M. aluminium alloys mostly depends on the oxides or adsorbed gases present on the surface of the small particles [1–7], and can be very low if these surfaces are not correctly degassed before sintering. Generally, the surface of aluminium powder consists of Al-oxides or/and Al-hydroxides formed during atomization. Depending on the composition of the alloy and the thermodynamic conditions, various oxides originating from the oxidation of alloying elements may also appear.

An important contribution published by Kim et al. [6] concerns the identification of oxides present on the surface of P.M. alloy 7091 with 6.5 wt% of Zn and 2.4 wt% of Mg. The authors observed that the powder is initially coated with a wet Al(OH)3.
According to this paper, degassing at 520° induces two important phenomena:

1) Degassing dries the surface layer by liberating water from hydrates. It is well known that several Al-hydroxides exist and that an irreversible process may occur at different temperatures:

$$\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O} \rightarrow \gamma-\text{Al}_2\text{O}_3$$

2) The liberated water may react with metals (Al or Mg) to form Al$_2$O$_3$ amorphous film or MgO crystallites:

$$3\ \text{H}_2\text{O} + 2\ \text{Al} \rightarrow \text{Al}_2\text{O}_3 + 3\ \text{H}_2$$

and

$$\text{H}_2\text{O} + \text{Mg} \rightarrow \text{MgO} + \text{H}_2$$

During hot pressing, dry Al-oxide and MgO crystallites facilitate the break-up of the surface oxides and promote a better homogeneous distribution of the broken-up product in the alloy. The mechanical properties of the aluminium alloy seem to depend greatly on such a distribution.

In another way, several authors have studied the degassing conditions of a cold compacted sample. In order to analyse the chemisorbed gases or the dehydration process of the surface, Morgan et al. [5] used a mass spectrometer analyser and presented ion intensities versus temperature for H$_2$O, CO, CO$_2$ and H$_2$. The hydrogen spectrum was characterized by two peaks, located at 350 and 390°, respectively. As mentioned by Morgan et al., these results are in good agreement with those published by Arbuzova et al. [4]. Hydrogen formation can be attributed to a reaction between metal and water liberated by the dehydration of oxides. These authors also pointed out a partial evaporation of zinc and magnesium at 480°. The optimal degassing temperature of such a compacted sample was determined at 400° just after hydrogen liberation. A correlation between hydrogen content, oxygen content and the ductility of the material has recently been established by Meunier [7].

In fact, the published papers concerning the optimal degassing temperature are not always in good agreement and the mechanisms proposed for hydrogen formation are not always clearly determined. In order to obtain more details about the mechanisms, we have made some temperature programmed desorption (TPD) experiments in an activated atmosphere (D$_2$O). The aim of the present investigations was to attempt to determine the origin of the hydrogen, the influence of the environmental gaseous water on the chemisorbed species, and the nature of the gas included in the powder.

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