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TEMPERATURE PROGRAMMED DESORPTION AND THERMOGRAVIMETRIC STUDIES OF OUTGASSING OF ALUMINIUM POWDER

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ABSTRACT

In order to obtain high quality powder aluminium alloys, it is necessary to optimize the degassing of the powder before sintering. By means of the temperature desorption technique and the thermogravimetric method, water and hydrogen evolution has been studied on a 7091 aluminium alloy. Concerning hydrogen formation, experiments confirm the part of water vapour in the environment and the part of water liberated by dehydration of alumina on the reactionnel process.

I. INTRODUCTION

The mechanical properties of powder metallurgy aluminium alloys are largely influenced by the surface states of atomized droplets [1-3]. The surface of aluminium powder generally consists of Al-oxides and /or Al-hydroxides [4-6]. During the thermal treatment for consolidation, water vapour may be liberated from hydrated aluminium oxide and may react with metal to produce hydrogen [7-9]. In such cases, the evolution of hydrogen induces cluster formation in the sintering material and the quality of the alloy largely depends on this phenomenon. To obtain a high quality, it is necessary to optimize the degassing of the powder before sintering. Such a process implies understanding of the desorption mechanism of H₂ from the surface of the powder. The aim of this work is to study in detail hydrogen evolution from the material by means of the temperature programmed desorption (T.P.D.) technique and the thermogravimetric method.

II. T.P.D STUDIES.

The apparatus consists of a quartz reactor chamber connected with a quadripole mass spectrometer. The temperature was raised uniformly at 2°C/mn. The powder was degassed (P < 10⁻⁵mbar) and heated to 500°C. During the heating treatment, the signal dN/dt = f(temperature) was recorded continuously (where N is either the water or the hydrogen gas concentration). Results are presented in figure 1 which shows a large water and hydrogen desorption. With reference to the hydrogen spectrum, it is interesting to note four peaks located at 230, 300, 365 and 390°C. The last peak is always followed by a partial sublimation of zinc. If the sample is cooled back with air one hour, degassed and reheat to 500°C, a large evolution in the hydrogen spectrum (figure 2) may be observed. The last peak at 390°C is the only one to regenerated ; consequently, the first three hydrogen peaks represent an irreversible phenomenon without any relation to the ambient water vapour. In order to confirm such a hypothesis T.P.D experiments have been carried out under activated water pressure (figure 3). In such a condition, H₂O is substituted by HDO or by D₂O on the surface of the sample. However, we note no deuterium evolves below 350°C during the first three evolutions of hydrogen. Alternatively, the last evolution at 400°C, essentially represents D₂ or HD. It follows that the water of constitution in the

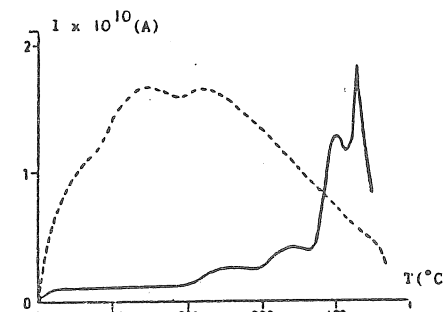


Fig.1 - T.P.D. curves of H₂(-) and H₂O(---) during the first thermal treatment.

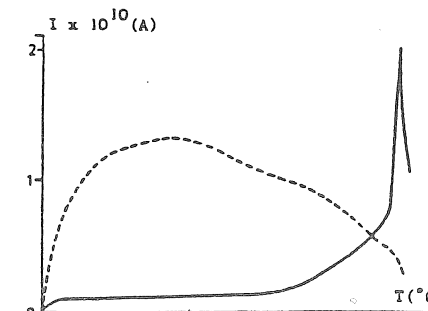


Fig.2 - T.P.D. curves of H₂(-) and H₂O(---) during the second thermal treatment

Al-hydroxides cannot be substituted by D₂O and that D₂O may react with the metal during the partial sublimation of the alloy, such a result implies a break up of the oxide larger during the sublimation.

III. T.G.A. RESULTS.

In order to obtain quantitative informations about the water temperature programmed analyses have been performed by mean of thermogravimetry. Results illustrated by figure 4 show two successive weight losses of the sample heated up at 2 or 20°C/mn and continuously evacuated ; the first may be explained by water and hydrogen liberation. The sublimation of alloying elements may be responsible for the second. Chemical analysis of the sample confirms such an hypothesis. It is also important to notice that sublimation only occurs when a specific quantity of water has been degassed. It follows that this phenomenon occurs at a temperature value which depends on the heating rate and therefore on the degassing kinetic process . Similar experiment has been carried out with an argon atmosphere (100 mbars). A large weight gain then occurs (figure 5) which may be ascribed to an oxidation process. As the initial and residual oxygen pressure is too low to induce such a phenomenon, we suggest that desorbed water may have dissociated. In order to confirm this hypothesis experiments have been performed with a circulation of argon . Figure 6 shows that no sublimation or oxydation is observed at temperatures greater than 400°C. This implies that water molecules are slowly

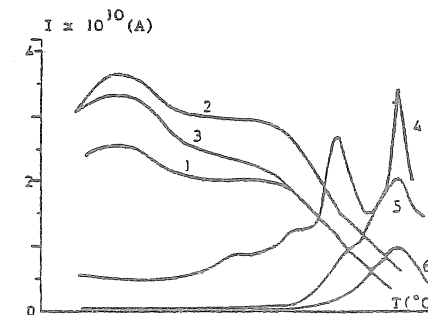


Fig.3 - T.P.D. curves of H₂O (1) HDO (2), D₂O (3), H₂ (4) HD (5), D₂ (6), during the first thermal treatment.

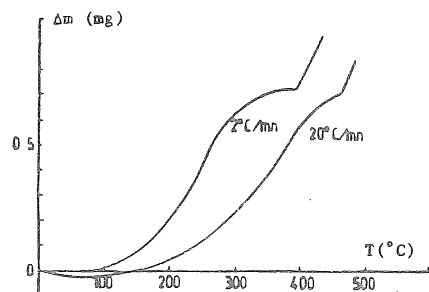


Fig. 4 - TGA curves under vacuum (10^{-4} mbar)

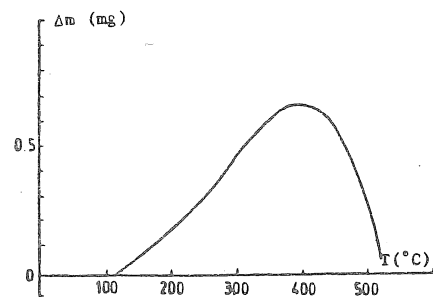


Fig. 5 - TGA curve under an argon pressure (100 mbar)

removed from near the powder by the gas flow. T.P.D. experiments have been carried out in a similar condition. Hydrogen spectra are presented in figure 7. The first three peaks are shifted at the high temperatures and the fourth peak which is directly connected to the water vapour concentration does not appear in dry air or in argon gas circulation. Furthermore, no metallic condensation has been observed.

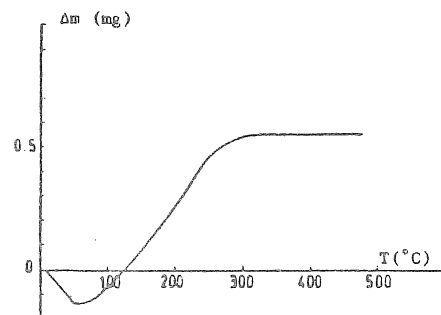


Fig. 6 - TGA curve under an argon flow (0,05 l/mn)

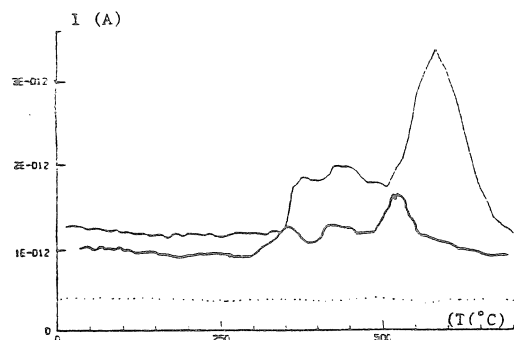


Fig. 7 - TPD curves of H_2 under an argon (≡) or a dry air (-) flow.

CONCLUSION.

Hydrogen formation on the 7091 aluminium alloy results from water decomposition at the contact of the alloying metals but two different mechanisms may occur.

- water is released as a function of time and temperature by aluminium hydroxides and may react with metal at the metal oxide interface.
- water is chemisorbed on the powder and the decomposition process is conditioned under vacuum, by the breakup of the oxide layer followed by an evaporation of zinc.

If water isn't evacuated by a pump or by a gas flow, it dissociates near the powder and is responsible for the catastrophic oxydation of the alloy.

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