The determination of oxides in magnesium based alloy AM60B

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INTRODUCTION

During the last few years the use of magnesium based alloys is increasing in many application fields, due to their low specific weight.
Usually these materials are controlled with elemental analysis and mechanical tests. In order to better understand their characteristics, we developed a method for the qualitative and quantitative determination of oxides.
In this paper we describe the results obtained testing some samples of the alloy AM60B.
Their composition (ASTM B93/B93M-94) is shown in table 1.

TABLE 1

Al (%)	Zn (%)	Mn (%)	Fe (%)	Cu (%)	Ni (%)	Others (%)
5.6 to 6.4	<0.20	0.26 to 0.50	<0.004	<0.008	<0.001	<0.01

SAMPLE PREPARATION

The samples used for the analysis are cylinder shaped (h=80mm, ϕ=5mm) and obtained from the heart of ingots of AM60B, with a milling-cutter in an inert atmosphere.
In order to reduce the possibility of formation of oxides during the sampling, the cut is performed while introducing in the hole a continuos flow of gas-oil.
This way of sampling assures the maintenance of low temperature without using water.
The cylinders are then cut in smaller pieces (h=5mm, ϕ=5mm) in a gas-oil bath. After cleaning them with petroleum benzine they are ready for the analysis.

ANALYTICAL PROCEDURE

The smaller cylinder is introduced in a graphite crucible and then heated in the furnace while the temperature increases constantly from 300°C to 2300°C. The instrument is maintained in a helium flow (inert gas).
The graphite of the crucible reduces the oxides of the sample according to the following reaction.

$$MeO + C \rightarrow Me + CO$$

The CO is then converted into CO_2 in a catalytic reactor, and detected by an IR cell.
A typical analysis graphic reports a series of peaks: each peak indicates one specific reaction of decomposition. During this reaction the formation of the gas (CO_2) reaches a maximum and then declines. The temperature at which the reaction takes place depends on the type of oxides being reduced.
Experimental data can be interpreted in terms of free energy of formation of oxides.
Fig.1 shows the relation between the free energy of formation of oxides and the temperature: the greater the free energy, the greater is the affinity of that element to the oxygen and hence the higher is the temperature of reduction of the oxide.

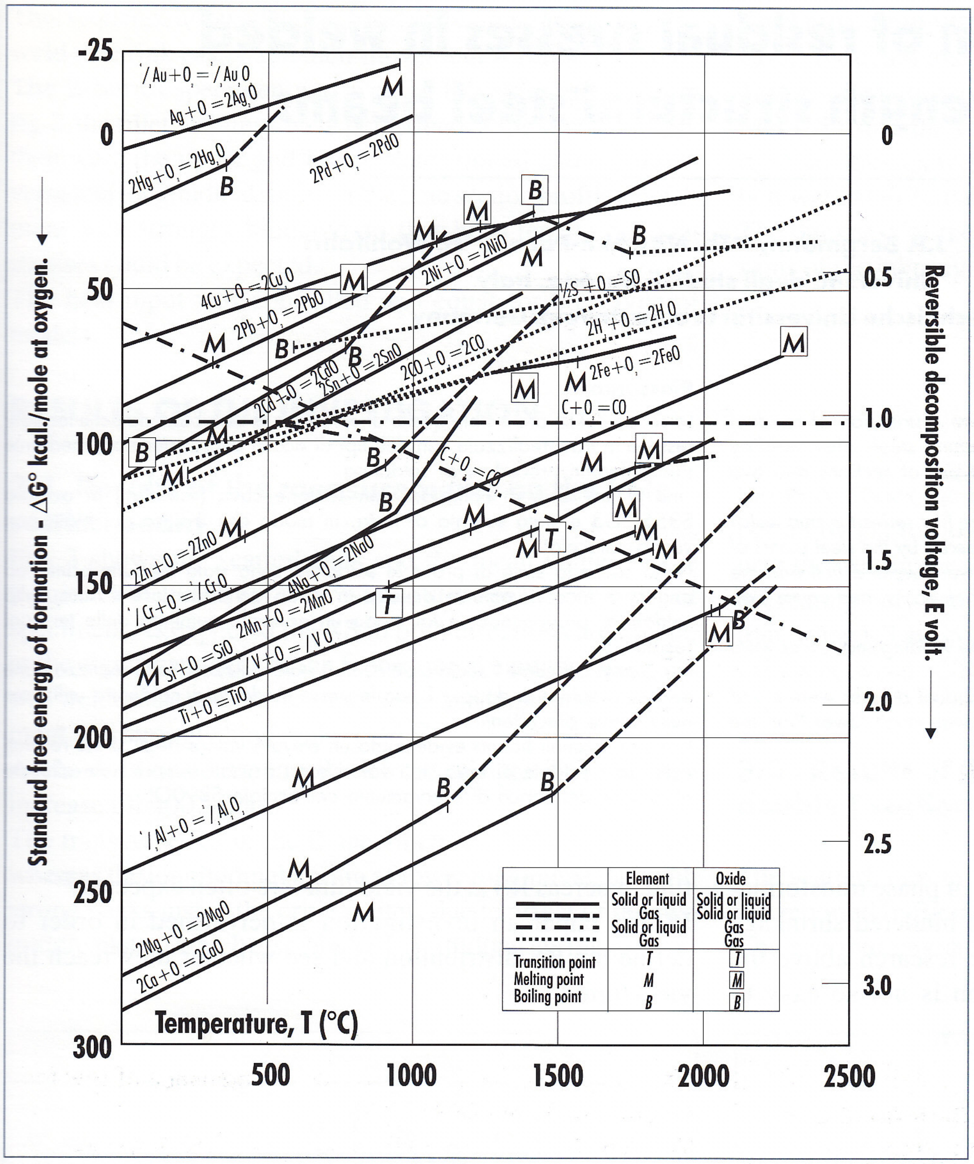

Fig. 1: Free energy of formation of oxides.

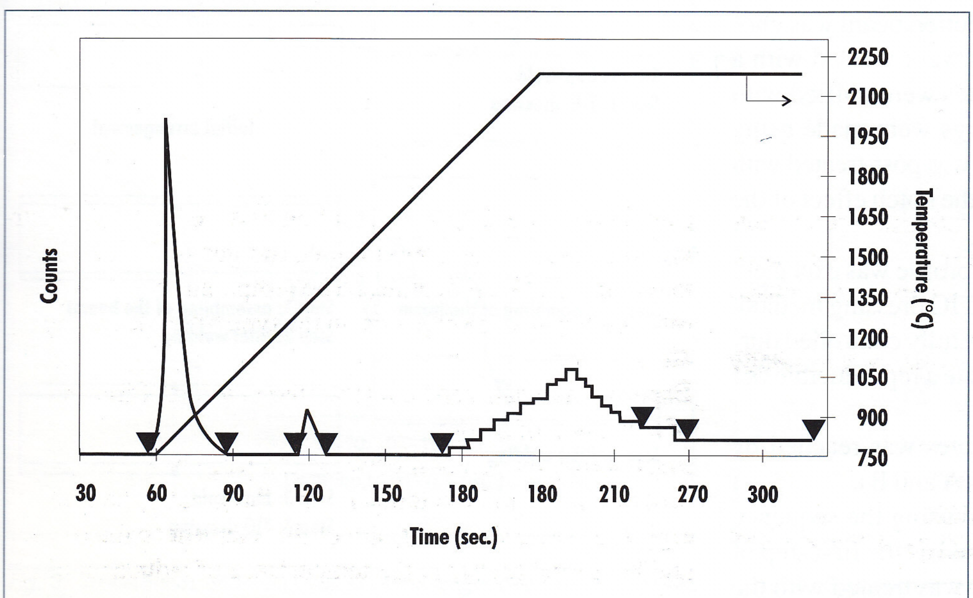

Fig. 2: Analysis of AM60B sample.

According to this interpretation, it can be possible to define the sequence of the oxides present in the sample (qualitative analysis). It is also possible to determine the relative amount of a single type of oxide (quantitative analysis).

Fig.2 shows an example of analysis of a sample of alloy AM60B: the time of the analysis is represented on the x-axes, the temperature of the sample and the intensity of the O_2 signal is represented on the y-axes. Three peaks can be observed: the first at a temperature below 750°C, the second one at 1400°C and the third at a temperature around 2000°C.

Considering the AM60B composition this last peak can be assigned to the decomposition of MgO or Al_2O_3 or to a combination of the two oxides.

The second one should be correlated with another oxide of a minor element of the alloy (Mn, Fe, ...), while the first peak is probably due to a superficial species of oxide, easily reduced as demonstrated by the low temperature of decomposition.

The content of oxides can be determined measuring the areas of peaks, then normalised to the sample weight.

CONCLUSIONS

We proposed in this paper a test method for the experimental determination of oxides content in magnesium based alloys.

This method has been applied to AM60B alloy and is now being tested with other materials.