



Sintering atmosphere critical for today's ferrous PM alloys

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The influence of atmosphere gas composition on the sintering of PM steels containing oxygen-sensitive alloying elements was the subject of several presentations at EURO PM2015. Some of the results of these researches are summarized in this report.

The sintering process has been a popular topic for presentations at international PM conferences for as long as one can remember. The EURO PM2015 conference in Reims, France, was no exception. Professor Herbert Danninger and his research team at TU Wien (the Technical University of Vienna) have been very active in this sector of PM research and were well represented at EURO PM2015.

For a number of years, low-alloy steel powders containing chromium have been in use for the manufacture of high-strength components, replacing previously used alloy compositions containing expensive nickel and copper. However, due to the high affinity of chromium for oxygen, sintering conditions have to be suitably modified, most commonly by the use of low dew-point nitrogen/hydrogen furnace atmospheres, typically in the ratio of 90% N₂ and 10% H₂. Over the years, many studies have been made on the process and the resulting material properties. Some of the latest work concerned with the interaction between atmosphere and compacted powder in the sintering furnace was reported in sessions at the EURO PM2015. One of the interesting features was the use of advanced analytical techniques.

Christian Gierl-Mayer and colleagues at TU Wien made a number of comparison experiments with plain iron powder and pre-alloyed low-alloy Cr–Mo steel powder to study the details of oxidation and reduction during sintering, as well as the resulting properties [1]. The metal powders employed were ASC100.29 plain iron powder and Astaloy CrM prealloyed steel powder, both from Höganäs AB. These experiments took a very interesting and illuminating approach. The sintering experiments followed the performance of intentionally pre-oxidized powders. In one series the base powder plus 0.5% graphite was pressed at 600 MPa with die-wall lubrication and then subjected to oxidation at 300 or 400 °C

for 30 or 60 min. For the second series the base powders were oxidized *before* compacting. The oxidation at 300 °C was performed in a drying oven, while the 400 °C treatment was in a tube furnace. Oxygen pick-up for the two types of powder was surprisingly similar, while on the other hand, as expected, the oxidation of the pressed samples was not homogeneous.

The oxidation treatments at 300 °C were followed by two types of sintering experiments: in the first series, sintering was done in a furnace at seven different temperatures from 700 to 1250 °C with a 90/10 N₂/H₂ atmosphere. In the second series, sintering was performed in a dilatometer coupled with a mass spectrometer, heating in pure hydrogen at 10 °C/min to 1300 °C, holding at temperature for 60 min followed by cooling at the same rate as the heating. The sintered samples were analyzed for carbon and oxygen, as well as the measurement of density, impact value, and hardness, plus examination of microstructure. The dilatometer/MS experiments provided insights into the reactions taking place during the heating cycle by measurement of specific gas species evolved. These tests provided a mass of data from which the authors concluded a number of interesting points.

The artificial oxidation at 300 °C resulted in an increase in surface iron oxides, and this behavior was similar for both the plain iron compacts and the Cr–Mo pre-alloyed material. Thus the presence of oxygen-sensitive alloying elements did not have a big effect on oxidation at that temperature. On the other hand, during the sintering experiments the behavior of the two types of material was markedly different. The iron–carbon material showed similar behavior in the step-wise sintering furnace experiments as in the dilatometer tests. The analyses of carbon and oxygen contents fit well with the dilatometer results. The microstructure, hardness and impact values followed the expected development with increasing temperatures, [Figs 1 and 2](#). For the Cr–Mo material, the

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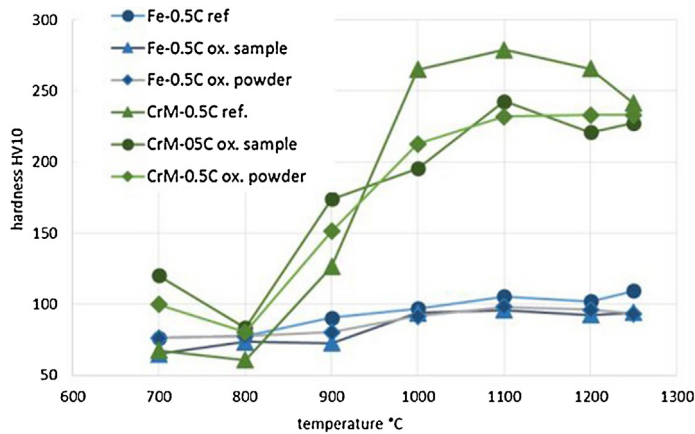


FIGURE 1

Hardness of differently treated PM steels sintered 30 min at varying temperatures in nitrogen/hydrogen. (After Gierl-Mayer et al.)

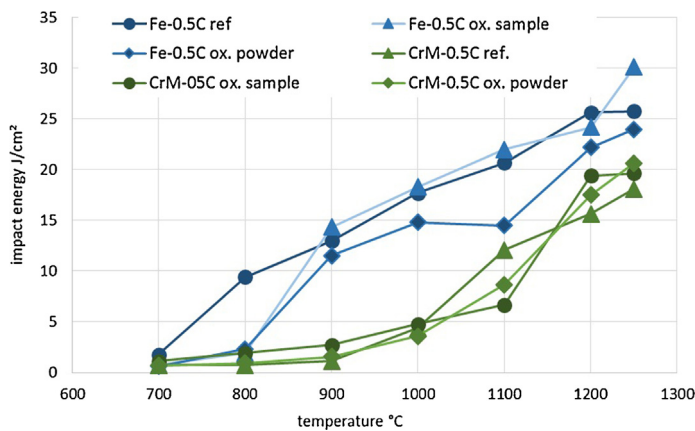
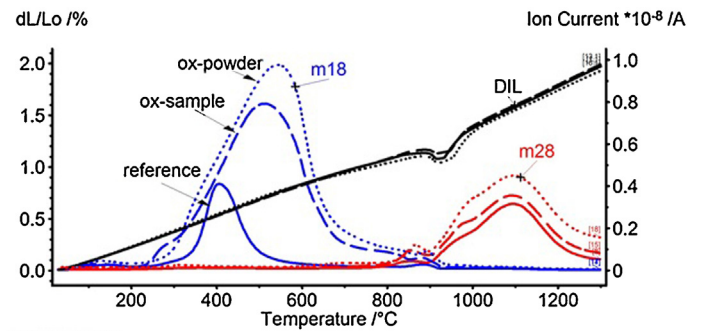


FIGURE 2

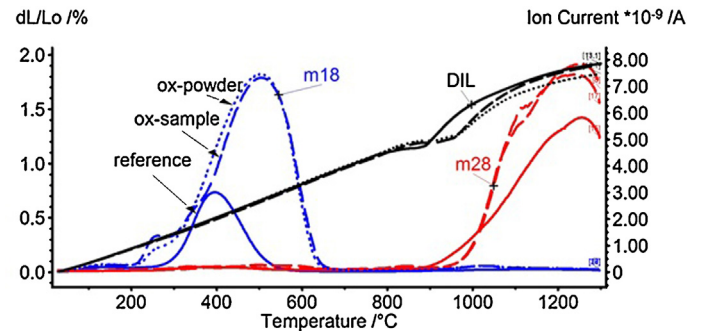
Impact energy of differently treated PM steels sintered 30 min at varying temperatures. (After Gierl-Mayer et al.)

results differed between the dilatometer and furnace sintering tests, suggesting possible internal oxygen transfer reactions (internal gettering). This was said to be more likely in the furnace experiments due to the rapid heating rate, versus the steady 10 °C/min rate of heating in the dilatometer (Fig. 3). The oxygen analysis showed that it took much higher sintering temperatures to reach 'acceptable' oxygen content in the artificially oxidized samples versus the non-oxidized reference material. This was consistent with much higher carbon loss and consequent lower hardness.

Following a similar quest, Hans Magnusson and co-authors at Swerea KIMAB and Höganäs AB, both of Sweden, focused on the role of carbon in sintering reactions with prealloyed Cr–Mo steel powder [2]. For experimental work, they employed another sophisticated analytical tool to monitor the surface reactions during sintering in N₂/H₂ atmosphere. Photoacoustic spectroscopy was used to measure specific species, H₂O, CO, etc., in the outgoing gases from the sintering furnace. (Photoacoustic spectroscopy is the measurement of the effect of absorbed electromagnetic energy, particularly of light, on matter by means of acoustic detection. It has become a powerful technique to study the concentration of gases at very low levels.)



(a) Fe+0.5C



(b) Fe-3Cr-0.5Mo+0.5C

FIGURE 3

DIL-MS versus temperature for differently treated PM steels, in pure hydrogen: (a) Plain Fe + 0.5% C, (b) Fe–3Cr–0.5Mo + 0.5% C. (After Gierl-Mayer et al.)

Test bars measuring 10 mm × 10 mm × 55 mm were prepared from Astaloy CrM (3% Cr–0.5% Mo) mixed with 0.5% natural graphite and 0.6% Kenolube lubricant. Powder was compacted at 600 and 800 MPa in a heated die to give green densities of approximately 7.0 and 7.2 g/cm³, respectively. The samples were sintered in a Kanthal laboratory furnace in 90/10 N₂/H₂, flowing at 3 l/min. Heating was either at 10 °C/min or 30 °C/min to a maximum of 1120 °C followed by isothermal holding for 30 min. The outgoing gas from the furnace was monitored with an INNOVA 1314 Photoacoustic Multi-Gas Monitor as a function of time and temperature. The results were compared with thermodynamic calculations made using Thermo-Calc software. Kinetic simulations of diffusion were made using DICTRA software. The photoacoustic spectroscopy (PAS) measurements during the sintering tests confirmed the low-temperature reduction of oxides by hydrogen, as well as the high-temperature carbon reaction forming CO. The higher heating rates (30 versus 10 °C/min.) were said to give more intense reactions, indicated by the higher concentrations of H₂O and CO in the atmosphere, Fig. 4. On the other hand, higher green density resulted in reduced gas reactions with lower concentrations of these gases. The thermodynamic calculations were in agreement with CH₄/CO and H₂O/CO transitions occurring at 700–800 °C in 90/10 N₂/H₂ and approximately 100 degrees higher in pure hydrogen, as seen in sintering tests previously reported by Chasoglou and Hryha [3], where pure hydrogen reacted with carbon to form methane. Under the same conditions, 90/10 N₂/H₂ atmosphere was seen to result in a barely measurable concentration of methane.

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