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Heat Treatment Solutions for Metal Injection Molding

The Metal Injection Molding (MIM) process is a widely known and applied technology for producing small metal parts. A great variety of materials and, consequently, approaches have been developed. The MIM process always requires a combination of polymer and metal powder. The polymer in this mixture has two purposes. On the one hand, it serves the rheological properties that are needed for injection molding. On the other hand, it provides the mechanical stability of the so-called green parts. The metal powder itself has no mechanical stability at all in that stage of the process.

The following step involves debinding of these parts, i. e. reducing the amount of polymer in that system. The type of furnace required for the debinding step depends on the polymer system used. One option is the so-called **catalytic debinding**. The polymer used is Polyacetal with a melting point above the debinding temperature. This polymer is decomposed in an acidic atmosphere to formaldehyde. Carbolite Gero offers the **Debinding Furnace EBO** (fig. 1) for this process step. The water-heated double walled vessel provides a very good temperature uniformity in combination with its sophisticated gas distribution. The design allows for safe and convenient handling of both acid and formaldehyde. The debinding process is not fully completed so the brown part may be transferred to a sintering furnace fur further treatment.

Figure 1: EBO furnace for catalytic debinding of injected parts

Another possibility is **thermal debinding** for which a greater variety of polymer materials can be used. For MIM the thermal debinding is done under an inert gas atmosphere in most cases. Process temperatures are higher than during catalytic debinding. The parts require slow heating rates to prevent them from cracking. Very precise temperature control directly at the parts is required. In addition, the off gases need to be lead to the gas system to avoid condensation on the parts which might cause carbon uptakes in the material. Vacuum, and therefore gas tightness, of the heated chamber in combination with precise gas control systems are required for high-quality parts and reliability of the process. This combination is provided by **Carbolite Gero's GLO system**. The flexibility of this furnace allowed us to develop a version that is designed for thermal debinding. After debinding, the brown part, which still contains a low amount of binder, is transferred to a sintering furnace. This binder has to be removed thermally. That is why at the beginning of the temperature program a slow heating rate is required to avoid cracks in the metal part. An additional holding step has to be introduced in the temperature program. Another parameter that needs to be considered is the gas type and volume. For low carbon steels like 316L pure hydrogen has to be used during the sintering step to avoid oxidation.

All these process-related requirements need to be considered. The **outgassing of the brown parts** needs to be handled by a special furnace design. A built-in retort avoids pollution of both the heating elements and the insulation. This helps to reduce down times due to cleaning steps and to minimize deterioration of the furnace's heating cassette. For the outgassing process, gas needs to be directed which can be done in two different ways. The **partial pressure mode** means working in a pressure that is slightly below atmospheric pressure. Gas is introduced from one side against a working pump. The **PDS sintering furnace** (fig. 2) has been developed for this approach. A different option is working in **atmospheric pressure**. Gas is introduced from outside the retort and finds its way into the retort where the brown parts are placed. The off gas of the brown parts is then forced into the gas outlet that is located inside the retort.

Figure 2: Furnace PDS for sintering of metal parts

The strong advantage of this approach is the possibility to switch the direction of the gas flow from inside the retort to outside the retort during the process. Hence, the obtained metal parts feature advanced properties. **Carbolite Gero's HTK furnaces can be adjusted to the specific requirements of the MIM process.**

The required parameters for successful manufacturing of metal parts depend mainly on the applied binder system. Some of the binder manufacturers in the market provide temperature programs that have been thoroughly tested with commonly used furnace types. Nevertheless, depending on the design of the parts, many aspects need to be considered which makes testing essential before the parts are released for mass production.

Testing is possible at places like the Karelia University of Applied Sciences in Joensuu (Finland) where the required infrastructure and the expertise exists to successfully develop a process.

In the field of Additive Manufacturing, MIM is a very promising manufacturing technique. Shaping is done by a 3D printer that melts the binder layer by layer. This extends the field of manufacturable parts to more complex shapes and new designs. All the following steps are then common knowledge with proven furnace designs.

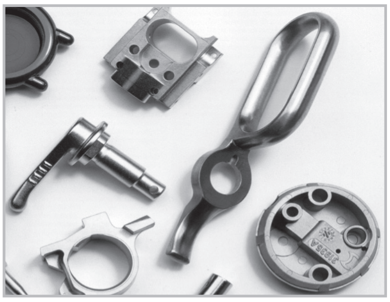

Figure 3: Exemplary parts produced by Metal Injection molding

Debinding and sintering of hard metals

Chamber Furnace HTK 600 GR/16-1G

For debinding and sintering, Carbolite Gero developed the **chamber furnace HTK 600 GR/16-1G**. This is a graphite furnace with a maximum temperature of 1600°C. It is equipped with a usable volume of 600 l, and a roots pumping unit which is used for **fine vacuum operation** as well as for **fast cooling** purposes. The fast cooling unit increases the availability of the furnace thanks to a cooling time of only 7 hours at full load. A single stage rotary vane pump is used as pre pump as well as for partial pressure operation of the furnace. The latter is especially important to give the user full freedom in adjusting the parameters to gain optimum diffusion properties for Cobalt (Co). The binder is collected in a condensate trap and is automatically released after each heat cycle.

Figure 4: HTK 600 GR/16-1G with fast cooling and fine vacuum pumping unit. The usable hot volume is 600 l, the maximum temperature is 1600 °C, achieved with graphite heating elements. Argon is the attached inert gas.

Application Hard Metals

The HTK can be used to fabricate saw tools for various kinds of application. Those could be wood working tools, rotating tools, window- or glass cutting tools etc. The small saw blades mainly consist of tungsten carbide (WC). A small amount of Cobalt (Co) and Titan (Ti) is also included. Mixed with polymeric binder (Paraffin), the powder is pressed into its final shape. Within a graphite furnace both debinding and sintering is performed. During debinding a controlled gas flow to protect the furnace is important. During sintering at 1450 °C max. the temperature must be controlled very precisely to preserve the small grain sizes of the carbides. With defined partial pressure atmosphere during the sintering step, Cobalt is diffusing towards the surface of the saw blades. This diffusion process allows to avoid a sputtering step afterwards, but requires high precision of atmosphere control in the furnace. Millions of tips for cutting tools are produced per day all over the world.

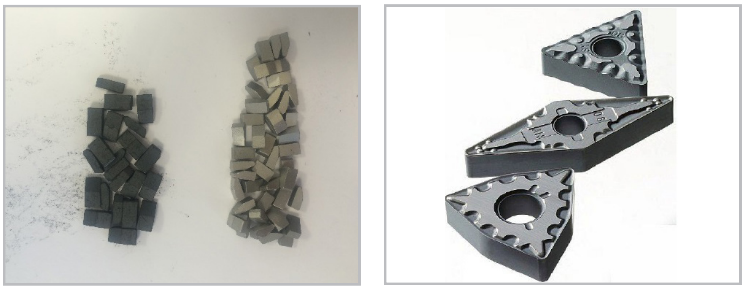

Figure 5: The produced WC parts prior to the heat treatment (left) and after debinding and sintering in the HTK (right)

Application details of tungsten carbide (WC) production

The different process steps

In a first step around 95% WC, 3% Co and 2% of Ta, Ti and others are available as powders with a defined grain size. The grain size is known from the raw material. Typical sizes are in the range of 0,8 µm. They are mixed inside a ball mill. Alcohol is added to prevent the powder from heating up during the mixing process which takes between 24 and 120 h. Depending on the varying amount of above materials, up to 16 different WC types are created by milling. A high Cobalt content makes the final WC sample softer; a low content makes it harder. Hardness is measured by the Vickers method (HV).

The next step is spray drying to get rid of the alcohol used during the mixing procedure. Together with alcohol a liquid mixture is created. Using hot nitrogen gas flowing over the liquid, the alcohol is removed and solid particles remain. Those particles are tumbled and filtered. Since there is still 2% of paraffin inside, the particles are shapeable. Now the WC particles, including the paraffin, are pressed into the final shape using molds.

The so-called green parts are ready for debinding and sintering which is done inside a graphite furnace in vacuum. During debinding, which happens between 280 °C and 320 °C, the paraffin is removed by evaporation. The binder is pulled towards the pumping unit through a condensate trap where it is condensed again. After that the paraffin can be easily released and recycled. During the debinding procedure, a temperature increase of the gas outlet can be observed. This is due to the high mass of the paraffin which creates an impact and heats up the tubings. At 650 °C a dwell time is introduced. Due to the large surface of the small samples, residual oxidation exists which is now removed by this intermediate dwell. It results in the release of CO_2. The next dwell is carried out at 1225 °C. The Co is melting and hence diffusing into the pores of the WC grains. This reduces the porosity and increases the final density of the material. The final sintering step is carried out at 1430 °C. By an intended drop of the temperature to 1200 °C and an adjacent increase to 1430 °C again, Co diffuses towards the surface of the material. This is helpful to avoid any sputtering steps after the sintering. Furthermore, it is necessary to have Co on the surface when the WC tips are brazed on circular saw blades. A pure WC surface, or worse a carbon-covered surface, cannot be brazed afterwards. Carbon-covered surfaces may result from inadequate debinding conditions.

Soldering is done after the heat treatment process in air by a CuAg solder. The WC is sharpened by grinding the final product.

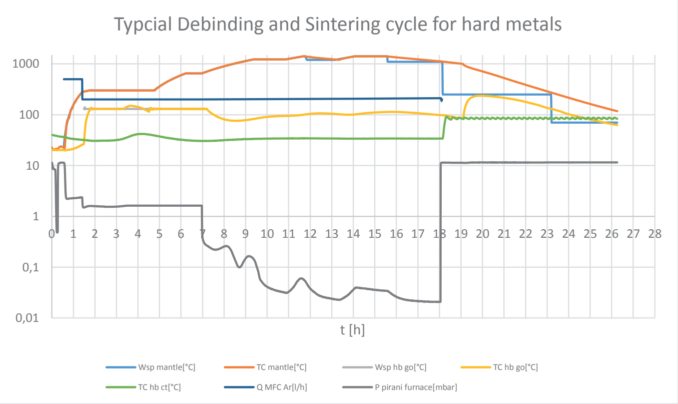

Figure 6: Typical Debinding and Sintering cycle for hard metals

Analysis of the sintered samples

The material is analyzed by XRF to determine the chemical composition of WC, Co, etc. X-rays are generated and focused to a mm spot size on the sample. The material's secondary X-ray's energy is characteristic for the element of the sample.

The hardness of the material is tested with the Vickers method. Density is determined by an optical microscope. It is aimed to be around 99.94 % of the final density.

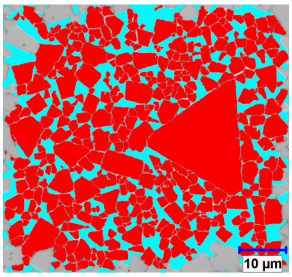

Figure 7: Optical microscopy of WC in a Co matrix

The sintered samples are analyzed in a SEM with EDX possibility. The "Z-contrast" is used to determine the surface quality. If carbon residues from an improper removal of the paffin are on the surface it can be detected by dark areas. Co has a brighter color. Co on the surface is highly desired to prevent sputtering steps after the sintering. The brightest color is generated by the W material because of its highest number of protons inside ("Z-contrast"). The pictures are generated with a primary electron energy of 20 keV. The determination of the surface quality is done by a comparison of the pictures.

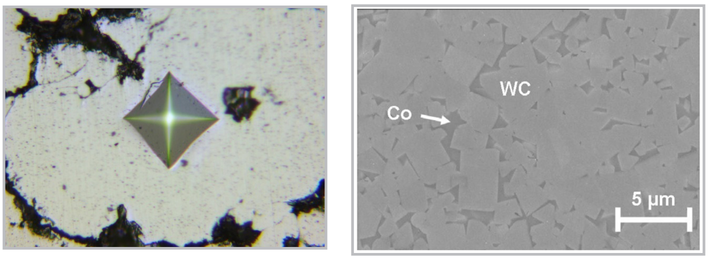

Figure 8: Vickers penetration into a sintered workpiece to analyse the hardness (left)

SEM picture of WC hard metal (right)