Application Note

MCQ Gas Blender 100 Series Application

Custom atmospheres' influence over sintered ceramic materials

Introduction: ceramic materials

Ceramic materials are non-metallic solid compounds characterized by a crystalline or semi-crystalline structure. Hard but brittle, strong in compression but weak in shearing and tension, characterized by good chemical inertness and high temperature resistance, ceramic materials can be both found in nature (mostly oxides) or artificially synthesized to create products with custom properties. Due to their specific mechanical, electrical and optical properties, ceramic materials represent a family of compounds used in a extremely vast field of applications.

MCQ Gas Blender 100 Series

- **Metallurgy**

Cemented carbides, also known as hard metals, are a group of ceramics materials characterized by a mixture of metal carbides embedded in a matrix of cobalt or nickel. Hard metals' physical properties make them especially suitable for the production of cutting tools and anti-wear components.

- **Electronics**

Both large-scale industrial production and R&D sectors exploit the wide range of characteristics related to ceramic materials. Compounds with different microstructure and/or composition are constantly developed in order to obtain electronic devices with enhanced performances. Semiconductors, dielectrics and ferroelectric compounds are fundamental components for every modern electronic application but ceramic materials are also promising substrates for the creation of semiconductors fuel cells (electricity production), solid state devices (pure oxygen production), superconductors (improved NMR devices) and many others.

The fabrication of ceramic materials is achieved with many advanced techniques, one of the most common is sintering.

Sintering is the techniques this application note is focused on due to the controlled atmosphere, required during the synthesis process to obtain satisfactory products. MCQ offers its Gas Blender 100 Series as the ideal instrument to create highly accurate custom atmospheres, easily adaptable to R&D projects aiming at the improvement of sintering results.

Sintering

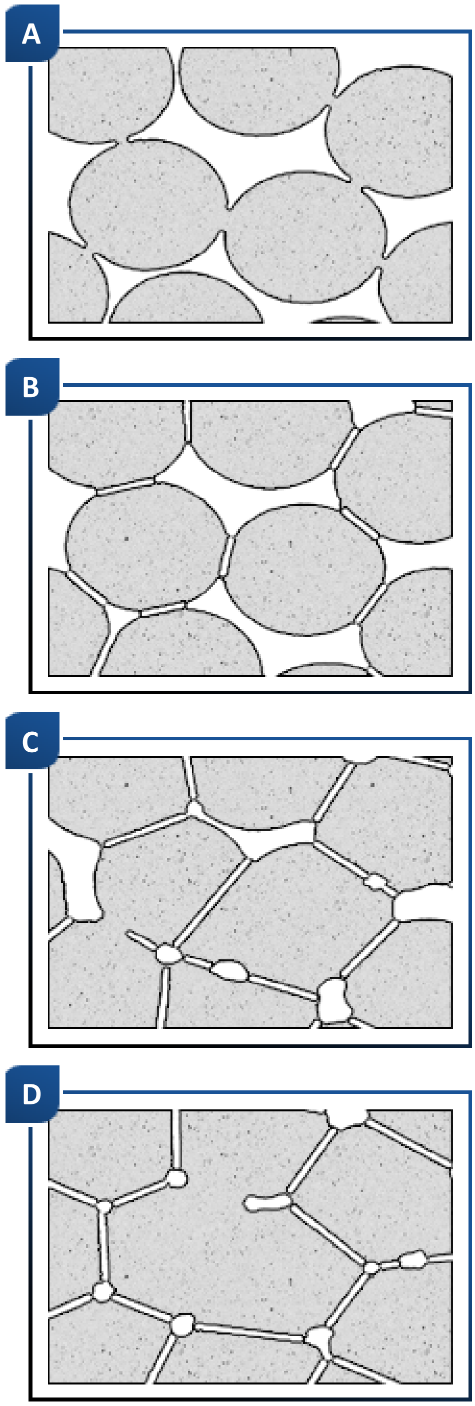
The basic principle of this technique is the possibility to create objects from powders, exploiting the diffusion phenomenon. The process consists in holding the powders inside a mold (properly shaped to obtain the desired object) and then heating the system to enhance the diffusion between the powder particles in order to achieve grains' coalescence. The chemical bonds that are established between the grains effectively create a new solid object from the starting powders. The heating process continues until the connections are considered satisfactory. This mechanism shares some similarities with the common "melting and solidifying" process, but the sintering technique offers some noticeable advantages:

- **Working temperature.** Reaching the melting point (m.p.), especially with tungsten or molybdenum based materials (m.p.>2600°C) is generally troublesome and often industrially unattractive. Sintering process occurs at significantly lower temperatures (around half of the m.p.), making the production process easier and more affordable.

- **Densification control.** The mechanism of coalescence between the powder particles leads to the formation of porous products. Densification, i.e. the gradual reduction of porosity that naturally occurs during the sintering process, can be controlled by managing the working parameters.

- **Grain size control.** The growth of grains, strictly connected with the densification, is another phenomenon commonly observed during sintering. Along with the porosity, the grain size strongly affects the chemical and physical properties of sintered ceramic materials.

The versatility of this technique allows to create materials with specific features by managing the process conditions. Quality and properties of final products are not only affected by working temperature but also by additives (compounds mixed with the powders in small quantities), working pressure and system



Sintering process

A. Grains rearrange their position, increasing adherence with other particles.
B. Particles' coalescence starts, generating the formation of necks between the grains.
C. Necks growth descreases the material porosity (densification).
D. Grains growth leads to an increased densification and material shrinkage.

atmosphere's composition. The working atmosphere is an especially critical parameter. Many sintering processes prove their effectiveness with standard air as working atmospheres [1] but pure gases or custom mixtures are often required to achieve the desired results.

Pure gasses

Pure gases represent the standard working condition for sintering. Nitrogen or argon usually replace air when the presence of oxygen can negatively affect the process outcome [2-3], while pure oxygen can be used to create extremely oxidant conditions. When severe reducing conditions are needed, sintering can be conducted under pure hydrogen [4]. Process conditions' strong influence on sintered materials leads many experiment to be carried out in different atmospheres, switching between various pure gases [5] to verify any possible improvement related with the changed working parameters. However, the choice of pure gases represent a limiting condition, particularly for R&D purposes.

Custom mixtures

Many literature works, during the past decade, proved custom gas mixtures' fundamental role to achieved specific advanced results. The use of custom atmospheres greatly enhances basic experimentation, increasing the range of working conditions and consequently widening the properties' spectrum of sintered materials.

- **Oxygen**

Oxygen is a key element for sintering experimentation, its presence drastically affecting the final product. The effectiveness and quality of mixed-conducting membranes (applied in gas/vapor separations and catalytic chemical reactions) and piezoresistive inks (commonly used for pressure sensor development) strongly depend on electrical conductivity, which can be easily modulated with the regulation of oxygen % in the sintering atmosphere [6,7]. The relative amount of oxygen also affects final density of sintered materials [8], influences the process of densification [9], alters the sintering temperature [10], and overall contributes to the parameters' optimization process [11-13].

Applications:

Common applications of various sintered materials: **A.** Industrial tools and components (tungsten carbide, WC). **B.** Heat elements (molybdenum disilicide, $MoSi_2$). **C.** Jewelry (WC). **D.** Solid oxide fuel cell for energy production (mixed oxides).

- **CO, CO_2, H_2**

Other gas compounds, whose chemical properties are exploited for sintering, are carbon oxides and hydrogen. Mixtures containing a combination of carbon dioxide (CO_2) and monoxide (CO) has been used for synthesis of spinels (base material for electric/acoustic devices) [14] or metallurgic powder preparations [15], while hydrogen has been used in combination with nitrogen or argon for various reducing mixtures experimentation [16-18].

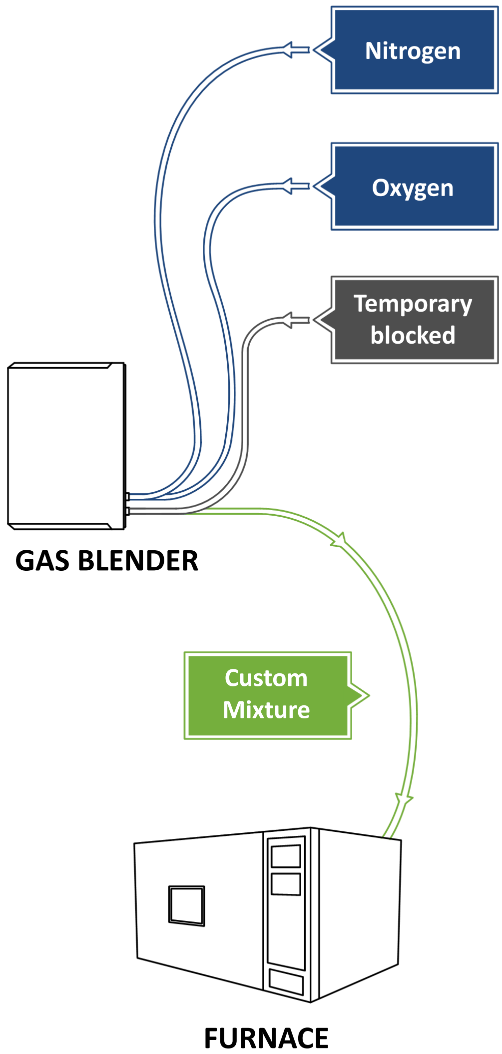
MCQ GB100

MCQ has recently developed the Gas Blender 100 Series, the ideal product for 3 components custom gas mixtures management. Designed following the “Lab in a box” principle, which replaces the standard bulky configuration of 3 Mass Flow Controllers connected with an external control unit, the Gas Blender 100 Series is a professional instrument that allow intuitive creation and instantly control of desired atmospheres with high precision (1% accuracy for each channel), high repeatability (0,16% of reading value) and the fastest response time for setpoint value changes (50 ms) available on the market.

Gas Blender setting and configurations can be easily manage with the MCQ Gas Mixer Manager software, bundled with the instrument and compatible with desktop and laptop working with any Windows operating system (starting from Windows XP).

Hardware configuration

An example of MCQ Gas Blender 100 Series hardware configuration is represented in the image alongside. The instrument works with dry, non-aggressive gases. The gas sources can be both pure or mixtures (in our example pure gases have been chosen for simplicity). The gas cylinders are connected to the instrument through 6 mm diameter tubes and a check valve is installed along each line as backflow prevention device. Each gas media is connected and controlled by a dedicated channel of the Gas Blender 100. Another 6 mm tube finally connects the instrument to the sintering furnaces in which the experiment takes place. A PC is connected to the Gas Blender through a simple USB connection. All the instruments features and the gas mixture properties can then be manage with the Gas Mixer Manager software.



Standard configuration:

Nitrogen and oxygen are blended togheter to create custom gas mixtures for the experiement. If the mixtures don’t require any additional component, the third channel can be temporary blocked. The gas mixture flows to the furnace, creating a the desired atmopshere for the sintering process.

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