DR.FRITSCH

FAST/SPS: The First Choice for Sputtering Target Fabrication

How FAST/SPS technology enables target manufacturer to meet customer requirements by improving target properties and allow a better positioning for the future.

FAST/SPS is a current activated, pressure-assisted densification and shaping process. In comparison to conventional sintering methods, in FAST/SPS the sinter material is heated by Joule's heating, facilitating much shorter sinter cycles, reduced grain growth, higher densities and tailorable microstructures. This is why our sinter presses are the first choice for the development and industrial-scale production of new and improved materials like (hard) metals, advanced ceramics, friction materials, heat sinks, thermoelectric materials and sputtering targets. The following article highlights the benefits of utilizing FAST/SPS for the manufacturing of sputtering targets namely high densities, small and uniform microstructure and all this at significantly reduced cycle times.

Introduction

Sputtering is a widely used method for the production of high-performance thin films and coatings for photovoltaic, solid-state-batteries, hard coatings and microelectronics. The starting point are monolithic to multi-element targets comprising of metallic or ceramic material. In a vacuum chamber the target is transferred into the vapour phase, as a result of bombardment from energetic particles generated by the plasma. The individual atoms from the target are deposited onto a substrate where they form a thin film.

While the thin film properties are generally optimized by modifying process parameters such as power, deposition rate and time, temperature and substrate property, one of the main prerequisites for the manufacturing of high-performance thin films is often overlooked or taken for granted: the quality of the sputtering target.

Alongside with characteristics of the starting materials (like purity, grain shape and grain size) the manufacturing route has a major influence on the quality and performance of the target. While there are several possible manufacturing routes, FAST/SPS (Field Assisted Sintering Technique / Spark Plasma Sintering) has proven to be the fastest and most versatile way to obtain high performance sputtering targets.

FAST/SPS is the short term for Field Assisted Sintering Technique and Spark Plasma Sintering. They are used as commercial names and are just synonyms for further developed hot pressing technologies, allowing high heating rates and temperatures up to 3.000°C. The term SPS is often used in academic literature, even though it is technically not correct. So far there were no sparks nor a plasma observed. Therefore, more and more manufacturers and also end users are using the term FAST. Other common names are for example Rapid Hot Pressing (RHP), Electric Resistance Heating, DC Current and Direct Current Sintering.

Why to choose FAST/SPS for target fabrication

In the recent years, FAST/SPS has attracted attention as a highly promising technique for advanced materials sintering. Reduced sintering time and high heating rates (>500°C/min) are the main advantages of FAST/SPS compared to conventional sintering. FAST/SPS thus has great potential to not only positively influence the properties of sputtering targets, but also to make them better controllable, as we will later see.

The manufacturing method is a decisive factor that determines the quality of the target and hence the performance of the resulting film and coating. Four quality characteristics directly affect the performance of targets:

- Density
- Grain size (and grain orientation)
- Uniformity of the microstructure
- Purity

Density is one of the most important factors in terms of performance. A higher density usually means higher deposition rates and less issues with outgassing. Film purity is also improved as gas inclusions and voids are minimized. The grain size and the uniformity of the microstructure affect the film uniformity on the substrate. Smaller, unimodally distributed and equal axed grains lead to a more uniform film. Besides, the grain size is inversely correlated to the erosion rate and consequently the deposition rate, meaning that smaller grain result in higher deposition rate.

A certain level of purity is necessary in order to guarantee desired film properties and reliability. The required purity is dependent on the final application and can significantly vary between applications. While for hard coatings purities around of ~99.5% (2N5) are sufficient, Integrated Circuit applications typically demand a purity of 99.95% (3N5). For semiconductors purities >99.999% (5N) are common.

Therefore, a high-quality target should have the highest possible purity, a high density, as well as fine and unimodal distributed grains (and a homogeneous grain orientation). With densities of up to 99.9% and homogeneously distributed grains with grain sizes even in the nanoscale range, FAST/SPS can meet these requirements almost perfectly, making it the number one choice for high quality target fabrication.

Comparison of main manufacturing methods

The following section describes and evaluates the main manufacturing routes for sputtering targets. Two main categories of fabrication can be distinguished, namely liquid phase processing such as casting and powder metallurgy (PM).

Casting

Simply put, casting is a relatively straightforward process. The material is molten and cast into desired shape. Major advantages lie in the simplicity of the process, high resulting target densities and low risk of contamination. However, casting faces two major problems: a) limited choice of materials and b) uniformity of the microstructure in the final target.

a) Refractory metals and the majority of ceramics have melting points of well beyond 2000°C, with the highest being close to or even higher than 3000°C (e.g. W, Ta, TiB_2, TiC). Casting of such materials is extremely difficult and if successful, they are very brittle. Casting is further limited by the solubility of the utilized materials – while for selected applications specific multi-element targets are demanded the elements might not be soluble in molten state. Hence, manufacturing is not feasible via casting.

b) The lack of homogeneity is mainly due to uneven grain growth and segregation. To overcome this, several additional techniques, like optimized cooling rates, casting closely above the solidus, (magnetic) stirring and/or vibrations during cooling (…) need to be applied. This makes the whole process more complex, costly, as well as time consuming.

PM

For the reasons above, the majority of sputtering targets are manufactured via PM. Its main advantage lies in the fact that only a fraction of the melting temperature is necessary in order to achieve full densification. Moreover, while not all compositions can be attained via melt casting, PM allows for convenient compositional adjustment by blending, mixing, doping and mechanical alloying. Another advantage in PM is that the microstructure can be easily adjusted by the selection of the characteristics of the starting powders.

Principally four PM methods are distinguished, namely conventional (or pressureless) sintering, conventional hot pressing (CHP), hot isostatic pressing (HIP) and FAST/SPS. The characteristics of the respective methods are summarized in Table 1 where they are displayed in comparison to each other.

All methods have in common that a material in form of powder or cold-pressed green compact is densified by diffusion processes in combination with temperature. The densification can be facilitated when a simultaneous pressure is applied. A distinction is made where the heat is generated and whether an additional pressing force is applied.

Conventional sintering

The simplest PM method is conventional sintering. Here, a cold-pressed green compact is sintered in an oven at high temperatures. No additional mechanical pressure is applied. In order to prevent contamination from the atmosphere, the sinter process takes place in a vacuum, inert gas or hydrogen gas. While the lack of a compressional force permits large target dimensions and large quantities at a low cost, it also prohibits complete densification and leads to long sinter cycles. Densities usually lie below 95% of the theoretical density. Long dwell times at elevated temperatures are needed in order to reach high levels of densification, which results in undesired large grain growth. Subsequent to the sintering process, thermo-mechanical treatment like hot rolling is crucial to enhance density and recrystallization annealing is necessary for microstructure refinement. The former introduces undesired textures in the target.

Conventional Hot Pressing

In CHP loose powder or a green compact is filled in a die and is subsequently sintered with a simultaneously uniaxial mechanical pressure being applied. A schematic drawing of the CHP set-up is given in Fig. 1a). The mechanical pressure (20-50 MPa) induces powder particle rearrangement and plastic flow at the particle

Table 1 - Comparison of different PM techniques for sputtering target fabrication.

	HIP	Conventional Sintering	Conventional Hot Pressing	FAST/SPS
Achieved densities	++++	+	++/+++	+++/++++
Cycle Time	++	+	+++	++++
Productivity	++	++++	++	+++*
Microstructure	++++	+	++/+++	++++
Material Versatility	++/++++**	+++	+++	++++
Ease of Use	+	++++	+++	+++
Dimensions relative to target dimensions	+	++++	+++	+++
Equipment cost	+	++++	+++	++
Summary	**19**	**22**	**23**	**27**

**Production is highly scalable with the machine range of Dr. Fritsch; **Starting powders for HIP are required to release only minimum gas contents in the vacuum sealed container*

contacts. This facilitates the sintering process, thereby leading to higher densities at reduced sintering temperatures and shorter sinter cycles. Sintering in a die, the final targets are near net-shape thereby minimizing the necessity for secondary machining.

In CHP the heat is generated from heating elements positioned concentric outside of the die. Another form of hot pressing is inductive hot pressing where the heat is produced via induction by an induction coil surrounding the die. This method can be considered as similar to CHP.

In CHP the heat is transferred to the sinter material via convection and conduction. This limits the heating rates to ~10-20 °C/min. In order to reach high densities long sinter cycles, usually >8 hours due to long cooling down times, accompanied with large grain growth, are needed. Major drawbacks of CHP are its low production efficiency and large grain growth during the process.

HIP

The general process of HIP is comparable to CHP, being densification under elevated temperature and pressure. However, instead of a uniaxial pressure, an isostatic gas pressure (most commonly with an inert gas like argon) of up to 200 MPa is applied. During sintering the powder or green compact is placed in a container and is subsequently vacuumed and sealed. The high isostatic pressure in combination with elevated temperatures lead to effective removal of the internal voids and allow target densities to reach nearly 100%. The required sintering temperatures in HIP are lower than in CHP and lead to smaller final grain sizes. Due to the isostatic pressure, the grain orientation is uniform. Drawbacks of HIP are the requirement for complex container manufacturing, high equipment cost and a large footprint of the HIP system. Additionally, cycle times are relatively long and usually between 2 to 8 hours.

FAST/SPS

FAST/SPS has a comparable set-up to CHP (uniaxial pressing under elevated temperatures), but instead of convective and conductive heat transfer from a resistant heated heating element , the sinter material and/or the adjacent die, are directly heated via Joule heating, thereby permitting very high heating rates. This is why FAST/SPS is also known under the name “rapid hot pressing” (RHP). Usually heating rates of >500 °C/min are reported for FAST/SPS, while the actual heating rate is dependent on sinter material, material of the die and the respective dimensions. So, in theory, heating rates >1000 °C are possible. Either way, such high heating rates lead to a set of advantages for target manufacturing via FAST/SPS. In short: high density, small grain size and tailorable microstructure.
A more in-depth explanation of the FAST/SPS principle is given in the following section.

Principle of FAST/SPS

FAST/SPS is a current activated, pressure-assisted forming process. The general set up is displayed in figure 1b). A FAST/SPS system is composed of a vertical single-axis pressure unit, a power supply providing direct

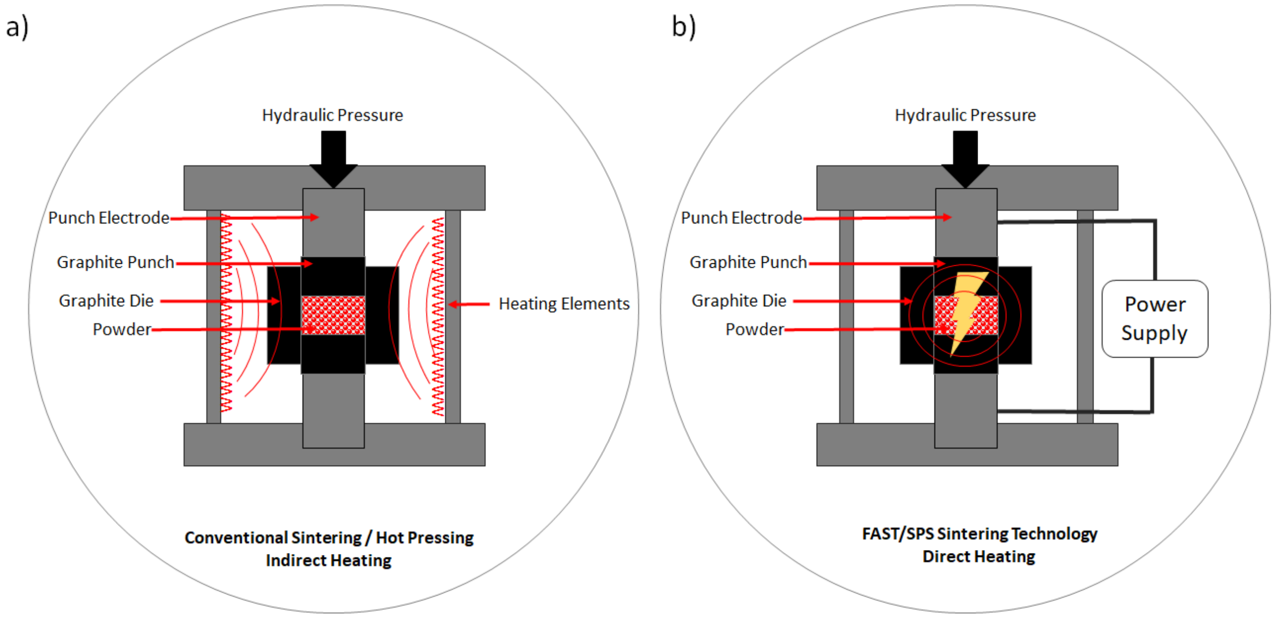

Figure 1 – FAST/SPS principle in comparison to CHP

current to the punch electrodes, and a graphite mold consisting of a graphite die and two graphite punches.

An electric current flows directly through the sintering mold and even through the sinter material if it is electrically conductive. The electrical resistance of the mold and the sinter material leads to a rapid heating. This is also referred to as Joule´s Heating. Compared to CHP which is displayed in a), there is no need to heat up the atmosphere. Temperature is consequently generated only where it is needed, allowing very short sinter cycles and high heating rates of even >1000 °C/min. The process can take place under vacuum or protective gas. Temperature measurement is done contactless by pyrometer or, in the lower temperature range, by thermocouples. Maximum temperatures achieved are up to 3.000°C.

Impact on sputtering target manufacturing

As specified earlier, sputtering targets require high density, small and equal-axed grains, a uniform microstructure and a high purity in order to deliver a high and even sputtering rate for the manufacturing of high-performing and reliable thin films. How does FAST/SPS help us to achieve this?

The main temperature-driven densification mechanism in sintering is grain boundary diffusion, while surface diffusion is the main grain coarsening mechanism. The onset of surface diffusion happens at lower temperatures, while grain boundary diffusion requires higher temperatures. A high-heating rate, as immanent to FAST/SPS, therefore reduces dwell-time at lower (grain coarsening) temperatures and high temperatures, where main densification takes place, are reached faster. The benefits are two-fold: a) the original grain sizes in the powder experience only negligible growth – they are "frozen" in the final target and b) sinter cycles are drastically reduced.

Additionally, potential agglomerations in the powder are rearranged and destructed under high pressures (>50 MPa), thereby further smoothening the microstructure.

FAST/SPS makes it possible to reach target densities close to 100% of the theoretical density, while simultaneously displaying a uniform and fine-grained microstructure. Both improve and ensure an even sputtering rate. Above densities of >95%, the pores are not interconnected thereby eliminating the risk of contaminants being absorbed by the target.

As presented in the introduction to this article, purity is a vital factor to ensure the required film properties and its reliability. The purity requirements are dependent on the final application of the thin film. In addition, the higher the purity of a powder, the less it is prone to form oxidic- or other unwanted phases. These can hinder even and optimum particle generation in the target. In conventional sintering, CHP and FAST/SPS, degassing and deoxidization, as a result of sintering in a reducing H_2-atmosphere or vacuum, can occur and boost the purity of the final target to some extent. Nonetheless, target purity is mostly influenced by the choice of the starting powders.

In addition to dictating the purity of the final target, the correct powder choice has a major impact on the sinter process itself. The sinter activity of the powder is largely dependent on grain size, grain size distribution and grain form (as a function of manufacturing process: e.g. from electrolytic processing, gas-/water atomisation or milling). These characteristics contribute to the apparent density and tap density. For high densities, generally small grain sizes are beneficial since they are characterised by large surface area which facilitates diffusion. However, a high surface area is usually accompanied by elevated oxygen contents, which has the opposite of the desired densification effect.

Another big advantage of FAST/SPS in target manufacturing is its potential for a wide range of materials. For the manufacturing of multi-element targets like e.g. Cu-In-Ga-Se (CIGS) or High Entropy Alloys (HEA), elements with differing crystal structures, different melting points, strength and hardness, need to be combined. While other techniques struggle, FAST/SPS can fully densify multi-element targets in short time and with tailored microstructure.

Market trends and continuous production

In the sputtering target market, there is an increasing demand towards larger and more productive FAST/SPS-equipment. A major factor that limits productivity in today's FAST/SPS systems, is that large workpieces (in today's standard up to 350x350mm), inevitably result in long cooling times. Besides, certain materials require long cooling times under pressure and inert gas to avoid cracks or material failure. Oftentimes these are about as long as the heating and holding times. In this time, the sinter press is merely used as an elaborate cooling device, thereby blocking it for any consecutive target batch, leading to a bottleneck with a stillstand-like situation.

Recent developments at Dr. Fritsch are tackling the challenges of larger workpieces and productivity in FAST/SPS systems. The image below is showing the latest innovation of Dr. Fritsch: the MSP-5 together with the MSC-5.

This machine separates the sintering cycle into two spatially separate processes. The MSP-5 is the sintering station. The MSC-5 is the cooling station. After the sintering process in the MSP 5, the workpiece can be shifted to the Cooling Unit MSC-5 under inert gas and vacuum, where pressure can be applied as well. For materials with long cooling times, this concept increases productivity significantly, up to two times as much.

The machine has 450 x 450 mm graphite electrodes and a pressing force of up to 5.000kN. It allows production of targets in the commonly used target dimensions as specified by Original Equipment Manufacturers (OEMs).

Figure 1 - The newly developed sintering press MSP-5 (left) together with the augmented cooling station MSC-5 (right).

Table 2 - Specifications for the MSP-5 and MSC-5.

	FAST/SPS Sinter Press MSP-5	**Cooling Unit MSC-5 (Optional)**
Total electric power	420 kVA	-
Max. Heating current	85.000 A	-
Pressing force	Max. 5.000 kN (Range 10 – 100%)	Max. 50 kN
Graphite electrodes (LxWxH)	450 x 450 x 100 mm Ø 450 x 100 mm	450 x 450 x 100 mm Ø 450 x 100 mm
Opening height	Max. 500 mm Min. 350 mm	Max. 500 mm
Temperature measurement	Thermocouple /Pyrometer at multiple positions	
Sintering atmosphere	Vacuum / inert gas	

Despite its high pressing force and sintering area, the MSP-5 is a compact and comparably lightweight machine. While existing FAST/SPS presses with only 4.000 kN pressing force are up to three floors high and require major construction adjustments of the building like foundation pits and ceiling breakthroughs, the MSP-5 can usually be installed on every normal production floor, thereby drastically reducing the cost of installation.

Learn more about Dr. Fritsch

Dr. Fritsch is the world market leader in FAST/SPS technology since 1953 with over 1.000 machines sold around the world. No matter the material - Dr. Fritsch FAST/SPS-sinter presses are the first choice for many applications.

On top of that, we are the only company worldwide that covers the whole value chain: FAST/SPS machines along with suitable raw material powders.

Our sister company Dr. Fritsch Metal Powders is specialized in retrieving, handling and production of raw material powders for a wide range of applications – including raw materials for sputtering targets.

If you want to find out more about how our FAST/SPS sinter presses can improve your sputtering target production, please feel free to get in touch with us.

This whitepaper has been co-edited by RHP-Technology GmbH (Seibersdorf, Austria), specialists in process and material development.

Dr. Fritsch Powder Shaping Technologies

Dieselstraße 8
70736 Fellbach / Germany

Telefon: +49-711-51832-0
Telefax: +49-711-51832-10

Internet: www.dr-fritsch.de
E-Mail: pst@dr-fritsch.de