NOUVEAU

D4.1. Dense (> 95%) interconnect fabricated via PM technology (FZJ)

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REVISION HISTORY

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1.0	13-01-2024	Martin Hilger / FZJ	Draft
1.1	14-02-2024	Christian Lenser / FZJ	Revised
1.2	27-02-2024	Marijke Jacobs / VITO	Final revision

EXECUTIVE SUMMARY

D4.1 aims to show the possibility of producing a dense (capable to separate SOC process gases) component of typical interconnect material (ferritic stainless steel, esp. Crofer22APU/Crofer22H) via a powder metallurgical (PM) route. Thereby, the foundation for various PM based processing methods, as well as the single-step manufacturing of the interconnect component with a coating (D4.4) should be created.

For that first study, the use of a state-of-the-art interconnect material, Crofer22APU (developed at Thyssen-Krupp and IEK-2 (FZJ)) should be combined with a modern time- and energy-efficient sintering method, Spark Plasma Sintering (SPS). A sintering study, focusing on the influence of the sintering temperature is conducted to determine a suitable parameter set to achieve a sufficient densification. Starting point for this study is the knowledge from former experimental studies on steel processing via SPS.

The definition of suitable sintering parameters for the stainless steel powder is crucial for the adaptation to the co-sintering with a coating material (or its precursors). By that, in a further study, a novel approach in the single-step manufacturing of a coated interconnect steel can be tested out.

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List of Abbreviations

Abbreviation	**Definition**
FAST	Field assisted sintering technology
PM	Powder metallurgy
SEM	Scanning electron microscope
SOC	Solid Oxide Cell
SPS	Spark plasma sintering

1 Experimental

1.1 Material

The manufacturing of a dense bulk interconnect is done with the state-of-the-art material Crofer22APU as powder (H. C. Starck). The powder's main particle sizes are ranging from 53 to 80 µm (supplier information). A characterization via dynamic light spectrometry showed characteristic values of 57 µm for d10, 76 µm for d50 and 100 µm for d90 with a monomodal particle size distribution. The material is used for the sintering experiments without any further treatment.

1.2 Field Assisted Sintering Technology/Spark Plasma Sintering

As a method capable of densifying ceramic and metallic components in short times (~min) and with a high energetic efficiency, the field assisted sintering technology (FAST) (also called spark plasma sintering (SPS)) is applied to densify the Crofer22APU powder. The FAST/SPS method is based on the current- and pressure induced rapid heating of a powder in a conductive, compressive die (most often graphite). A scheme of the technique as applied in this work is given in Figure 1.

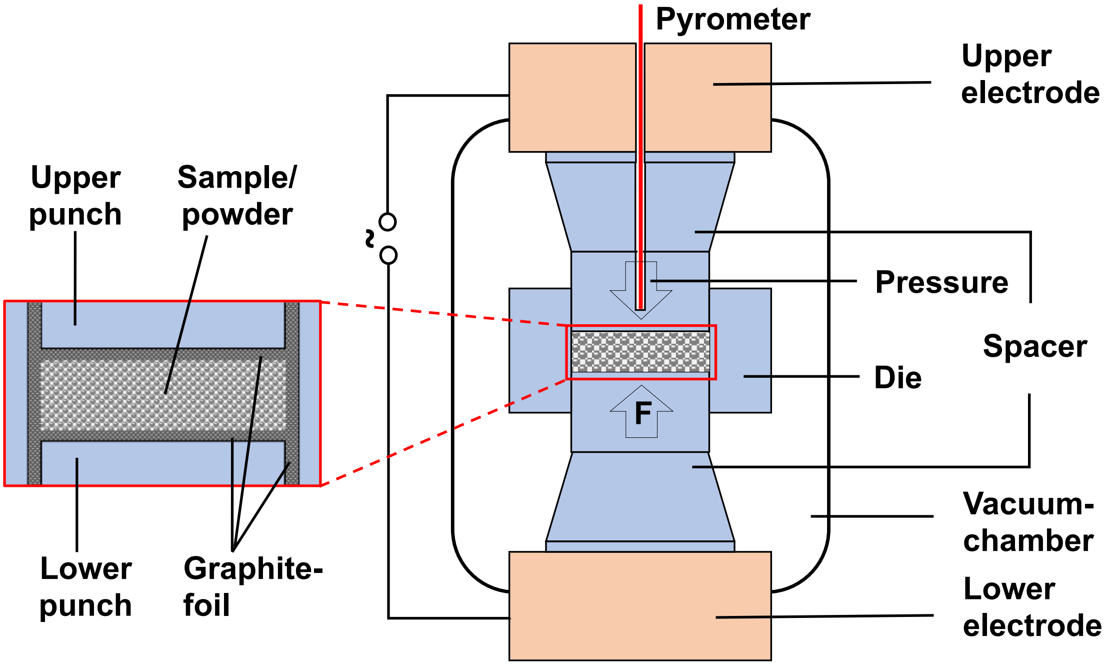

Figure 1: Schematic of FAST/SPS for the manufacturing of dense metal components, here sheets of Crofer22APU. Own graphic, derived from (Bram et al. 2022).

When using FAST/SPS, the grade of densification on the processing side is mainly influenced by the applied pressure, current and the generated heat (resulting in the sintering temperature), as well as the sintering time. Regarding the pressure, not only the pressure applied during the sintering but also during the pre-processing is essential for the final condition. In this experimental study, both, the pre-pressing as well as the SPS-processing was done at 50 MPa, while in other cases the pressures can reach up to 250 MPa (Guillon et al. 2014). Heating rates (for conducting materials, as steels) can range up to 1000 $Kmin^{-1}$ (Bram et al. 2022). In this study, throughout all experiments 100 $Kmin^{-1}$ was chosen. The dwelling time for all experiments was set to 5 min, while the cooling step at ~100 $Kmin^{-1}$ is connected to a continuous pressure reduction over 8 min to 0 MPa. The sintering temperature was varied in 100 K steps between 800 and 1100 K.

For each experiment, a powder mass of 10 g was pre-pressed at 50 MPa into a graphite die, lined with graphite foil. The dies are of cylindric shape with a diameter of 20 mm. After the SPS-process, the sintered pellets were removed from the die and residual graphite foil was ground off.

1.3 Characterization

The SPS-samples were characterized with regard to their density (as one key property of the interconnect is to separate the process gases, the fuel and the oxidant/air, from each other). The microstructural characterization was performed via scanning electron microscopy (SEM) of metallographically ground and polished cross-sections. The SEM images were analyzed qualitatively with regards to the development of the grain sizes and quantitatively via Fiji (image analysis software) with regards to the densification.

2 Results and discussion

Already when using a sintering temperature of 800 °C, at the chosen processing parameters, the generation of coherent pellets from Crofer22APU was possible. This was observable for all further samples. The cross-sections of all SPS-processed Crofer22APU samples, embedded in Epoxy, are shown in Figure 2.

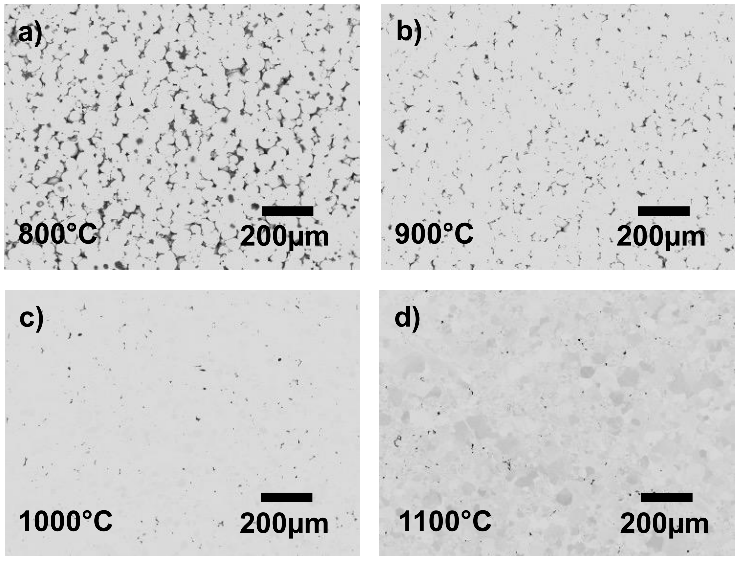

Figure 2: Cross-sectional SEM images of steel sheets (Crofer22APU) processed via FAST/SPS, depending on the sintering temperature, a) 800 °C, b) 900 °C, c) 1000 °C and d) 1100 °C.

In the microstructures it can already be observed that at a temperature of 800 °C, large fractions of isolated and connected pores remains. This is significantly reduced by increasing the temperature. The development of the density and porosity over the temperature is shown in Figure 3.

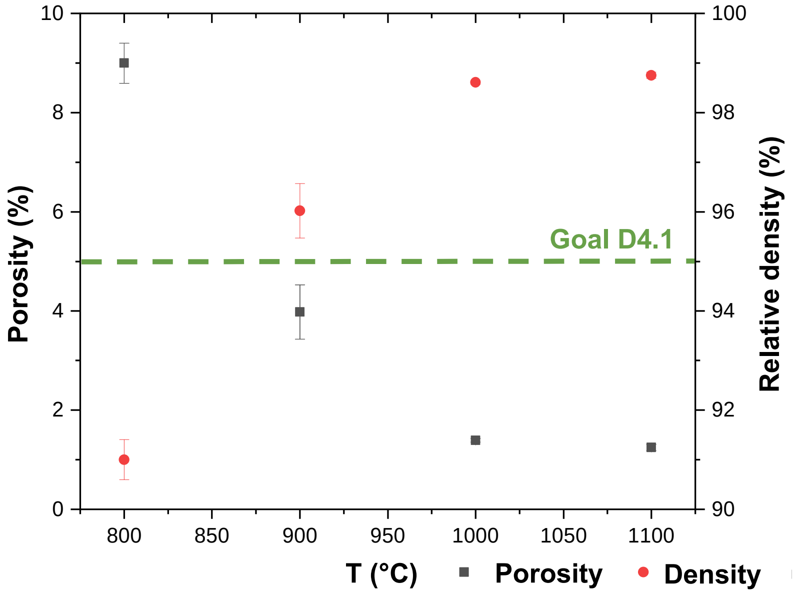

Figure 3: Porosity and relative density of steel sheets (Crofer22APU) processed via FAST/SPS, depending on the sintering temperature.

While the pore fraction is nearly the same for 1000 and 1100 °C (Figure 2 c and d), a significant increase in the main grain sizes is observable at 1100 °C due to increased diffusion activities. For all temperatures above 900 °C, the goal of a densification of >95% is reached (98.8% for 1100 °C). By that, the preparation for further studies with regards to the combination of steel and coatings and/or thermal post-processing is done.

3 CONCLUSIONS & NEXT STEPS

In this deliverable of WP4, the main goal of manufacturing a dense (>95%) sheet from a suitable interconnect material on a powder metallurgical route was reached. Therefore, a modern, time and energy efficient method, FAST/SPS, was used to sinter the state-of-the-art interconnect material Crofer22APU within minutes. A sintering study showed that densities above 98% were achievable for sintering temperatures above 1000 °C.

This is an ideal base for the adaptation to the investigation of co-sintering the interconnect material with a suitable coating material using the FAST/SPS method. As a coating material for this study, a state-of-the-art material that was already applied by various techniques, a $Mn(Co,Fe)_2O_4$ spinel, is chosen. The aim of this study will be to simultaneously sinter both components to a coherent compound with a strong adhesion, a high coating density and by that a suitable prevention of both, Cr-evaporation, and interconnect corrosion.

4 INDEX OF ALL FIGURES