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# MACHINABLE POROUS GOLD STRUCTURES FOR DECORATIVE APPLICATIONS MADE VIA SUPERSOLIDUS LIQUID PHASE SINTERING

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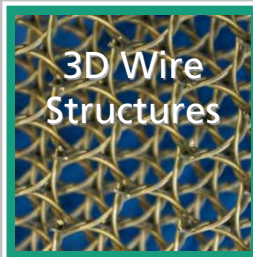
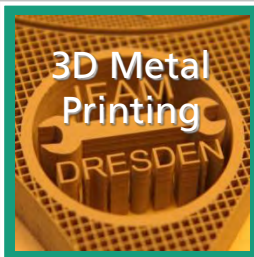
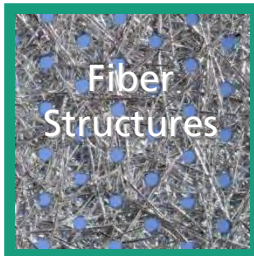
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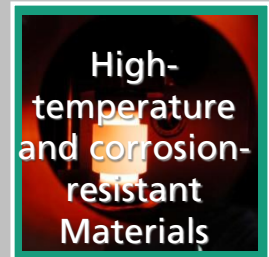
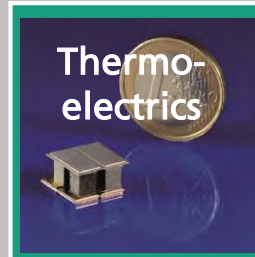
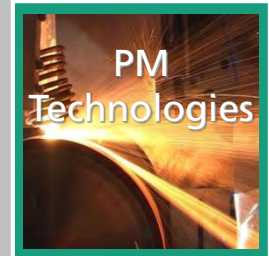
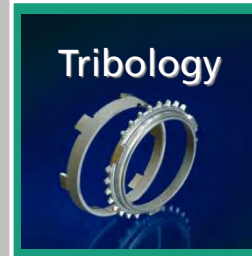
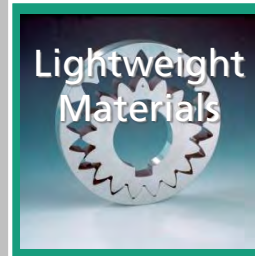
# Profile of Fraunhofer IFAM Dresden

## Fields of competence

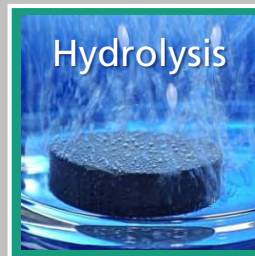
### Cellular Metallic Materials



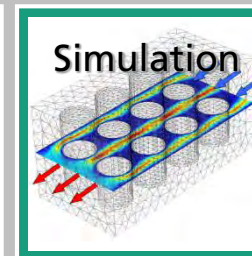
### Sintered and Composite Materials



### Hydrogen Technology

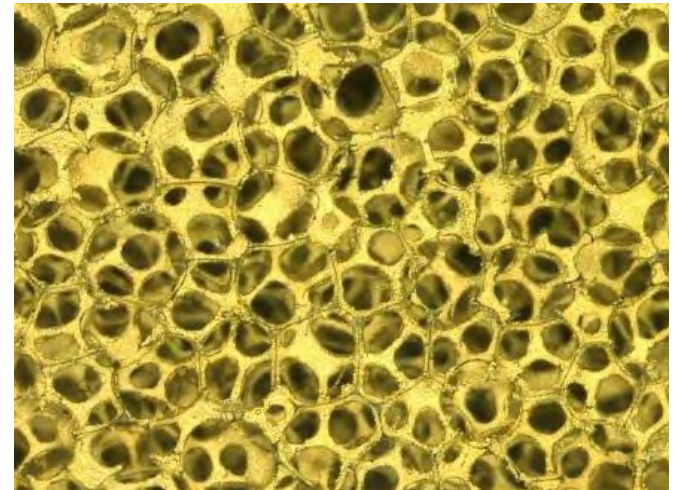


### Energy und Thermal Management



# Contents

- Motivation
- Sintering of 18 ct. gold alloys
- Sponge-type porous gold structures
- Fibrous porous gold structures
- Shaping of parts by high-speed milling
- Conclusions / Take-home messages



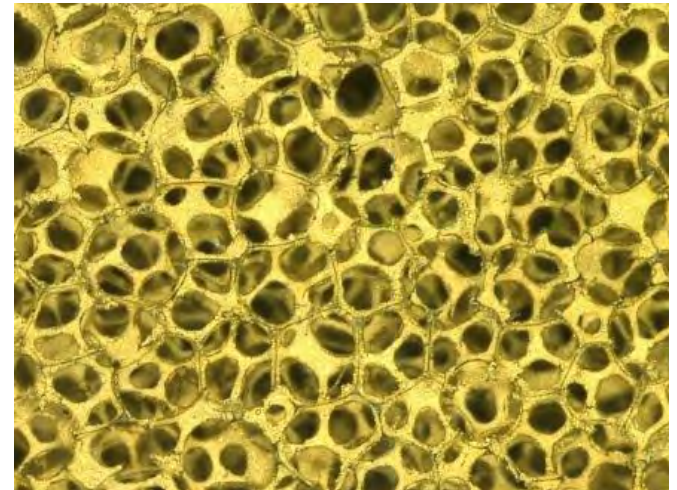
# Motivation

Feasibility study for the manufacturing of sintered 18 ct. porous gold structures. Goals:

- Cover a wide range of porosities
- Develop a milling route in order to produce precisely shaped parts

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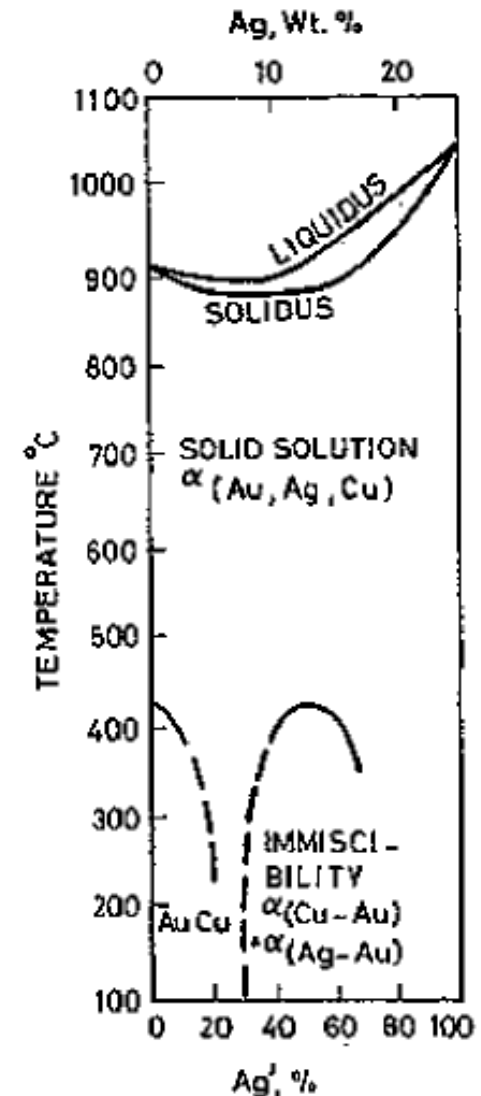
# Sintering of 18 ct. gold alloys

Main results of literature study:

- According to the literature, commercial sintering is done in solid state only and requires from 3 up to 24 hours of sintering time
- No publication on supersolidus liquid phase sintering was found
- The majority of the reported sinter atmospheres is composed of an inert gas (either N<sub>2</sub> or Ar) and 5 to 20 % H<sub>2</sub>
- The gas mass flow should be low in order to provide a constant temperature throughout the samples

# Quasi-binary phase diagram for 18 ct gold alloys

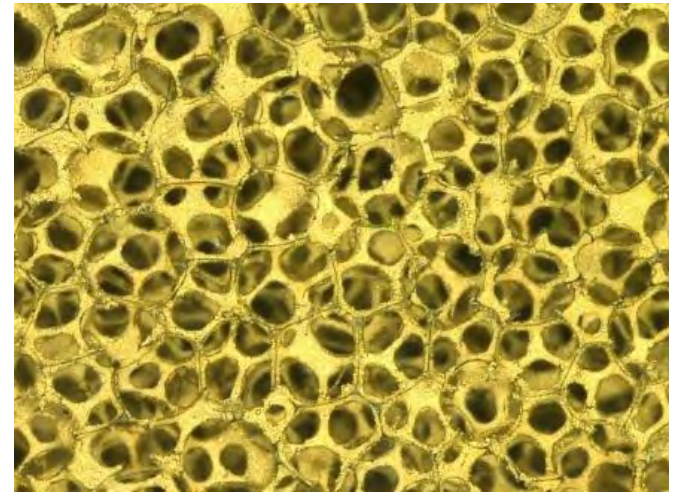
- Temperature interval of roughly 30 K between the solidus and liquidus temperature →
  - Sufficient for controlled super-solidus sintering with approx. 20 to 30 vol% of liquid phase
  - Should result in a drastic reduction of the required sintering time
  - Should result in larger sinter necks between the single particles / fibers
- ➔ Therefore, supersolidus liquid phase sintering was preferred over solid-state sintering in order to produce the desired porous structures



Source: A. S. McDonald, G. H. Sistare: The Metallurgy of Some Carat Gold Jewellery Alloys- Part I - Coloured Gold Alloys. Gold Bulletin 11(1978)11, 66-73

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- **Sponge-type porous gold structures**
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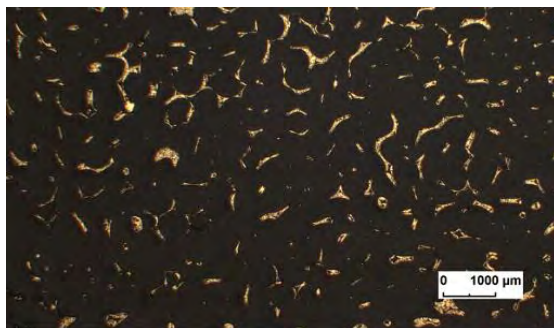
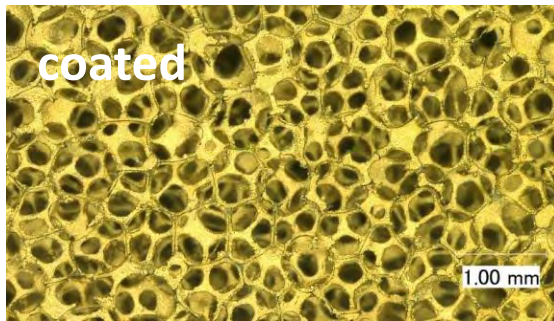
# Manufacturing of sponge-type gold structures via the Schwartzwalder process

- Reticulated polyurethane foam is coated with a metal powder containing slurry
- Organic constituents are burned out
- Remaining metal powder particles are sintered  
→ purely metallic sponge structure with hollow struts remains

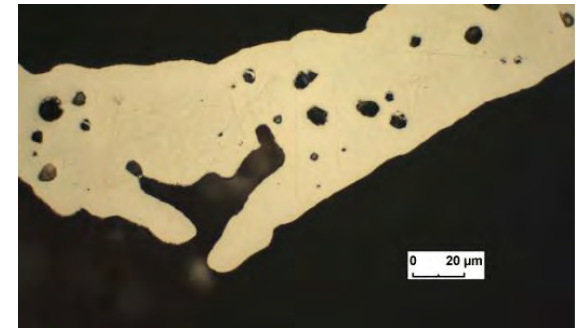
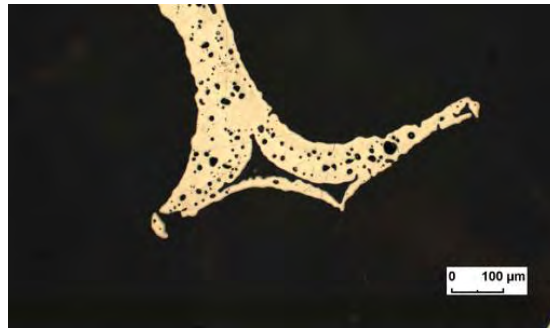
Patented by Schwartzwalder and Somers in 1961  
(US 3.090.094)



# Foam - Results

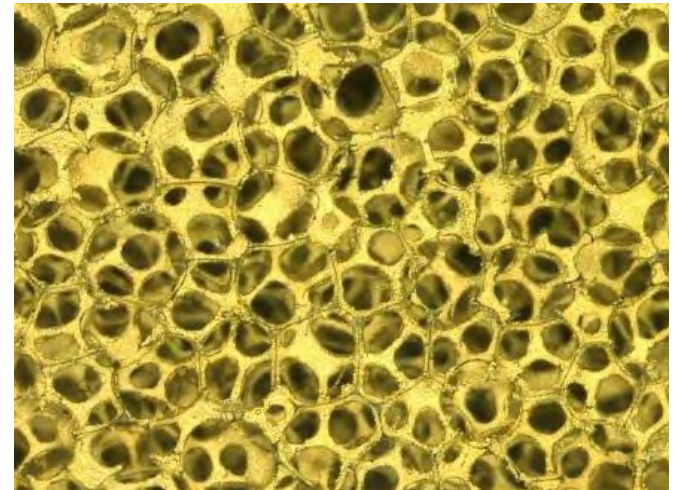


- 75 Au 12.5 Ag 12.5 Cu gas atomised powder with median particle size of 9.3  $\mu$ m
- Template: reticulated PU foam with 60 ppi
- Slurry based on PVA
- Debinding scheme with a maximum temperature of 400 °C
- Supersolidus liquid phase sintering at 888 °C for only 15 minutes

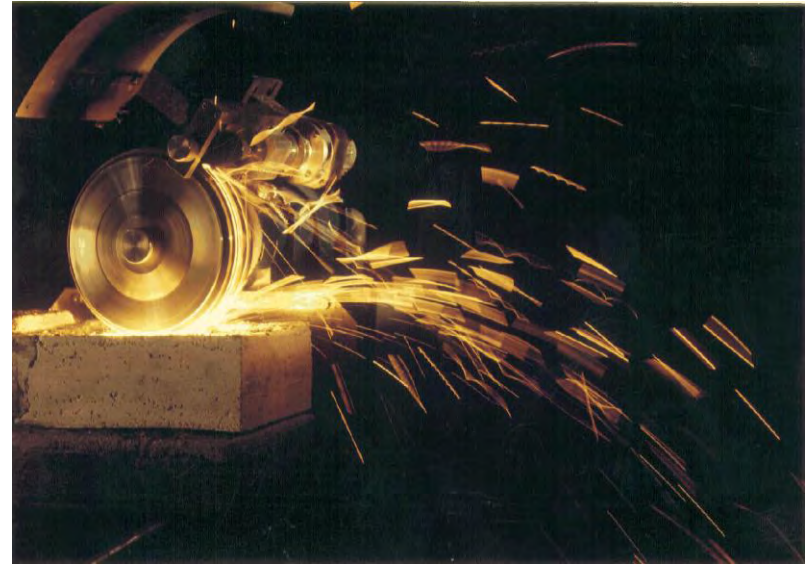
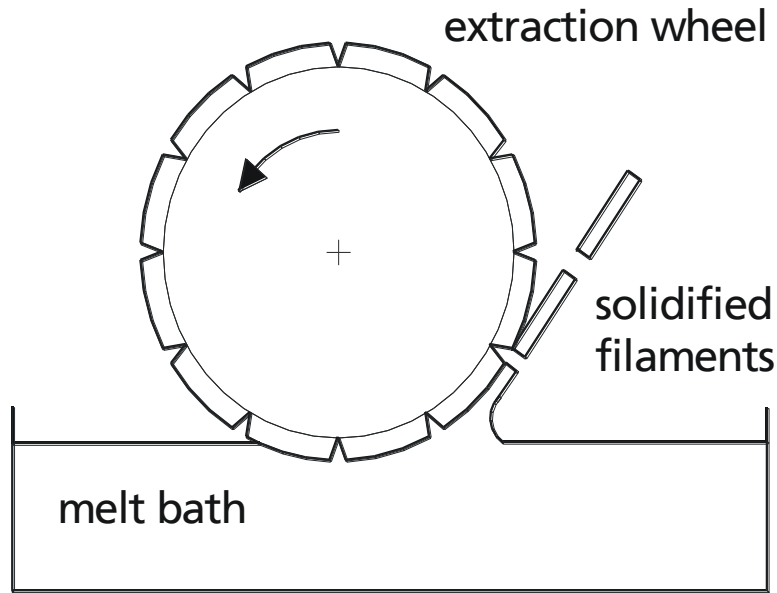


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# Manufacturing of short metallic fibers at IFAM Dresden



Principle of melt extraction

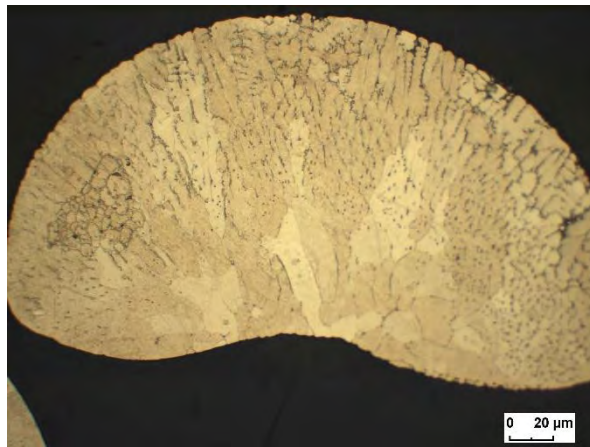
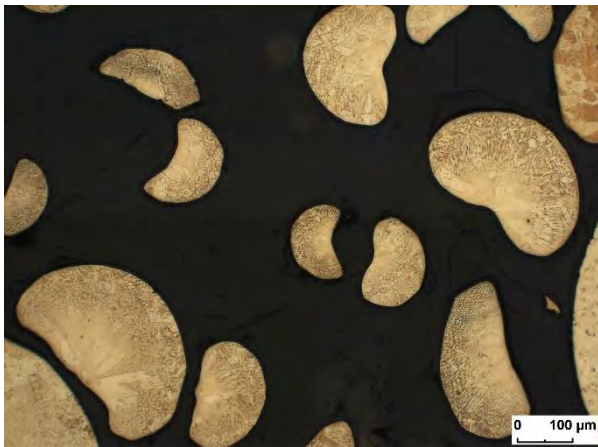
# Fiber production by melt extraction



- All common metals and alloys (Al, Mg, Cu, Fe, Ni, Ti, Au, Pt, etc.)
- Intermetallics (FeAl, NiAl, TiAl)
- Fiber diameters ranging from 50 to 250  $\mu\text{m}$
- Fiber length ranging from 3 to 50 mm

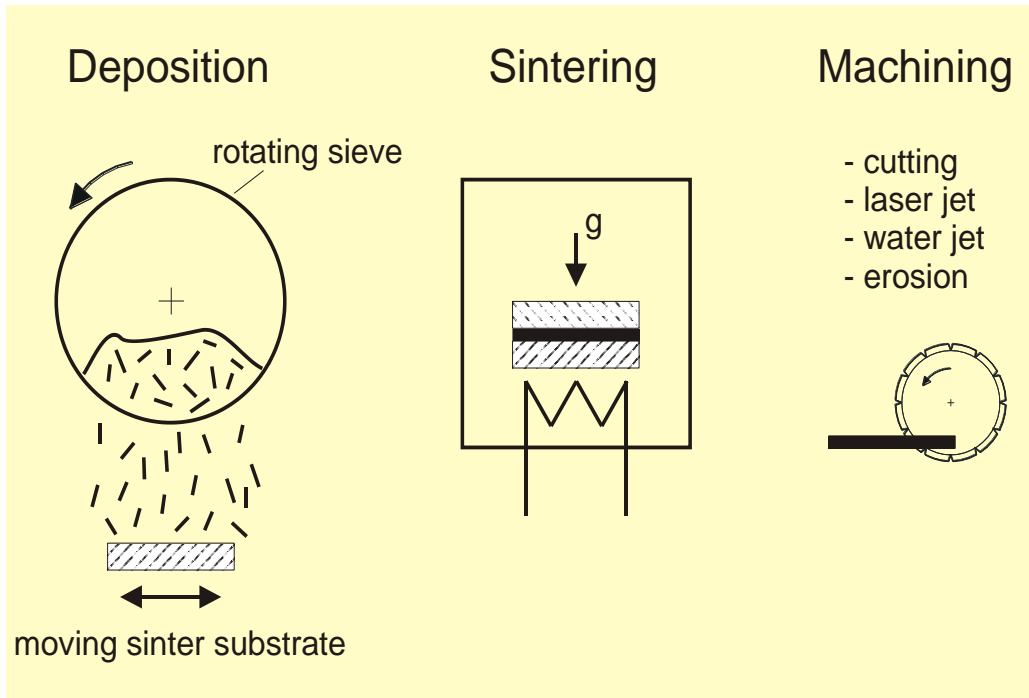
# Properties of melt extracted gold fibers

Sample No.	Mean Equivalent Diameter (Metallography) ( $\mu\text{m}$ )	Standard Deviation of Diameter (Metallography) ( $\mu\text{m}$ )	Mean Length (mm)	Standard Deviation of Length (mm)
V644/1	95.7	27.1	5.0	1.2
V644/2	83.3	35.6	5.1	1.1
V644/3	68.9	18.9	5.2	0.9



Etched cross sections of gold alloy fibers V633. Etching was carried out in aqua regia for two minutes

# Manufacturing route for sintered short fiber structures



- Plates up to 400 x 400 mm<sup>2</sup>
- Open porosity (50 to 95 %)
- Pore sizes of approx. 10 to 250 μm
- Graded structures possible

# Unsintered fiber deposit

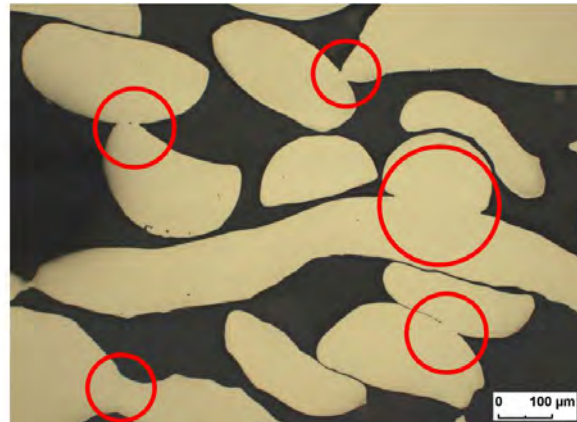
The tap density of the fibers was determined to be roughly  $3.4 \text{ g/cm}^3$ . Assuming a density of  $15.4 \text{ g/cm}^3$  for bulk Au750 N3, this corresponds to a relative density of 0.22 or a porosity of 78 %, respectively → some compression during sintering required to reach porosities of 60 % and below





# Comparison of sub- and supersolidus sintering

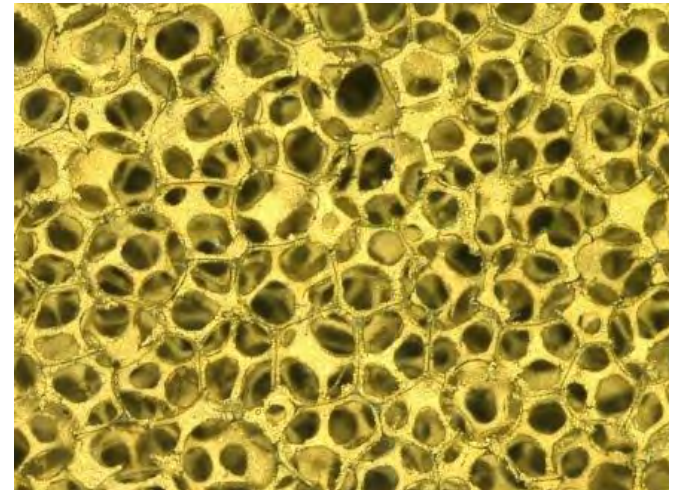
Sample No.	Sinter Conditions	Mean Hardness HV 0.1	Standard Deviation HV 0.1
490040_0017	825 °C / 180 min	265	10.2
490040_0018	890 °C / 10 min	281	3.7



Supersolidus liquid phase sintering results in pronounced sinter necks and higher hardness with less scatter across the sample

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# Sintered and machined red gold fiber structures

40 mm x 20 mm x 10 mm



dia. 10 mm center hole



Typical milling conditions: rotational speed 28,000 1/min, maximum cutting speed 2,000 m/min, cutting depth for finishing: up to 0.5 mm. Prior to milling, the fiber structure is filled with a liquid which is then solidified and removed after machining has been finished

# Conclusions / Take home messages

- 18 ct. porous gold structures were manufactured successfully by supersolidus liquid phase sintering
- The dwell time for sintering could be reduced down to 10 to 15 min, whereas conventional solid-state sintering for dense parts usually takes from 3 to up to 24 hours
- Supersolidus liquid phase sintering also resulted in a more homogeneous microstructure
- High-speed milling successfully demonstrated on porous gold parts
- The newly developed route provides a fast and easy way to produce almost arbitrarily shaped porous 18 ct. gold parts for decorative applications

# Thank you for listening!

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