

Additive manufacturing of Al_2O_3 - Nb composite granules based on paste extrusion (15th Freiberg Refractory Forum 2024)

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AM of Al_2O_3 - Nb refractory composite granules

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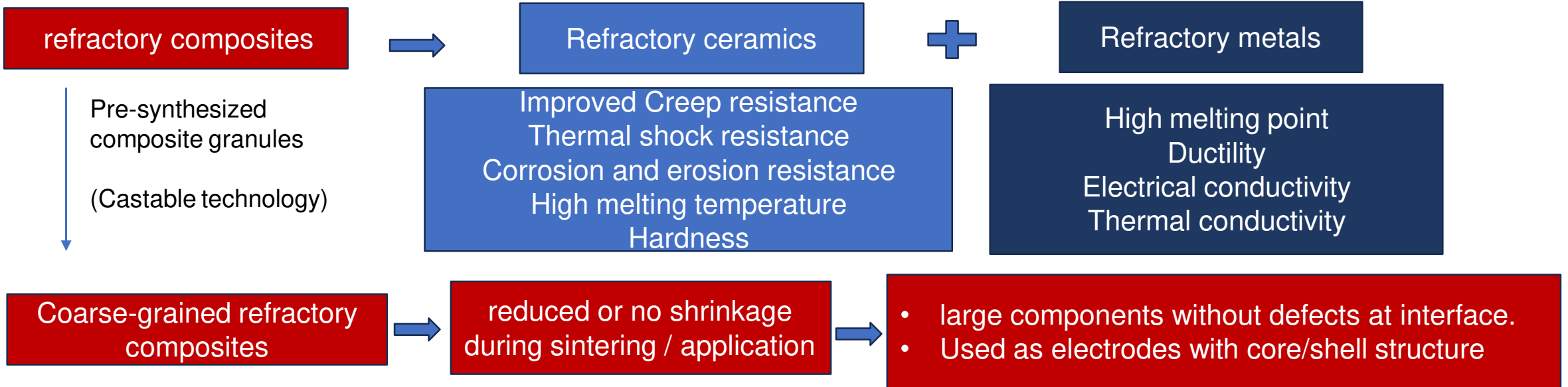
Supervisor: Dr.-Ing. Tilo Zienert

Supervisor: Prof. Dr.-Ing. habil. C. G. Aneziris

Introduction

Background of the study:

(Two step approach):



Objective :

Additive manufacturing technique based on paste extrusion



Paste formulation

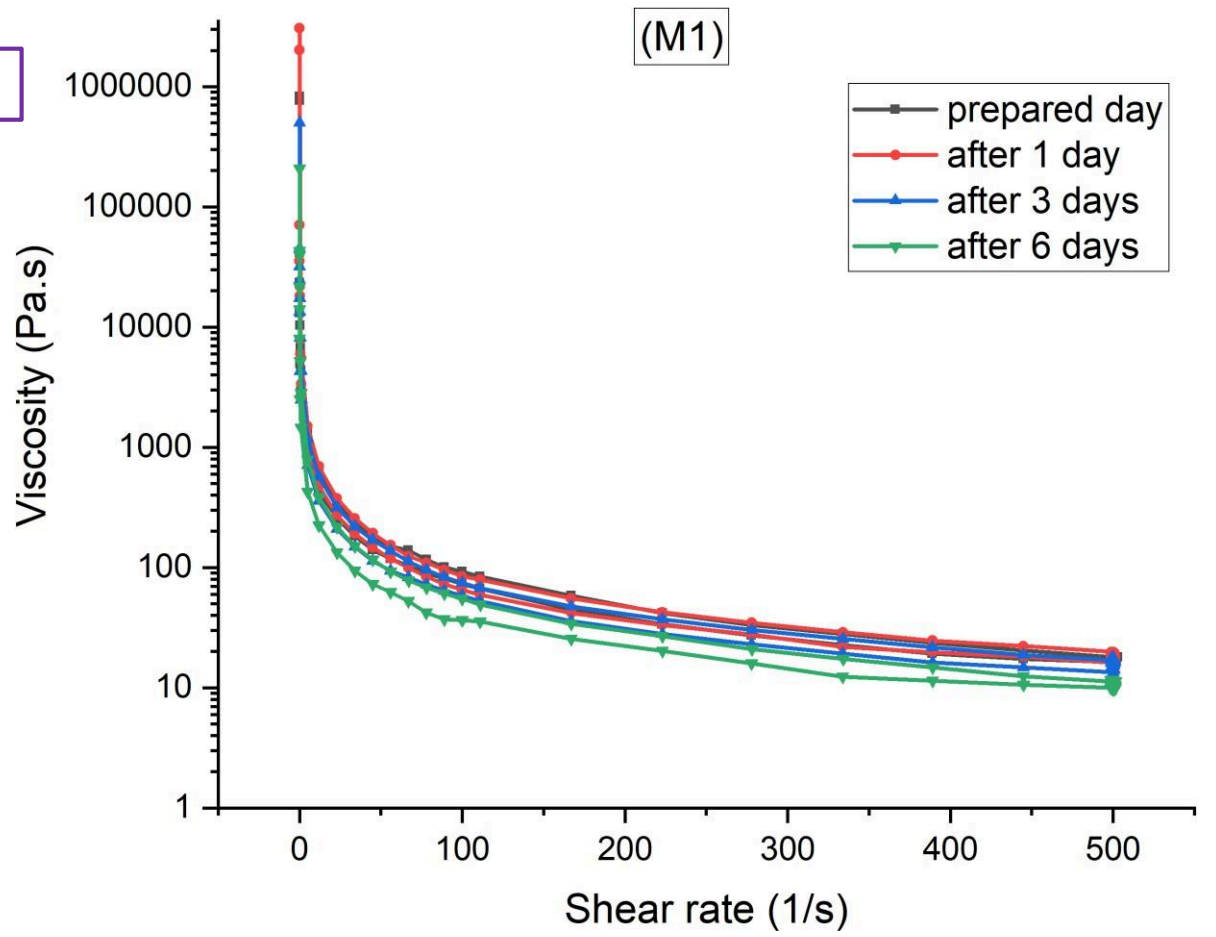
Al₂O₃ layer (100 vol%)

d_{50} -5.53 μ m

Al₂O₃ (70 vol%) - Nb (30 vol%) layer

d_{50} - 30.8 μ m

Materials	Vol %	Vol %
Niobium	-	28.69
Alumina (CT9FG)	95.64	66.95
Cellulose (KP 3039)	2.51	2.51
Xanthan powder	0.85	0.85
Oleic acid	1.00	1.00
De-ionized water	46.48	45.00



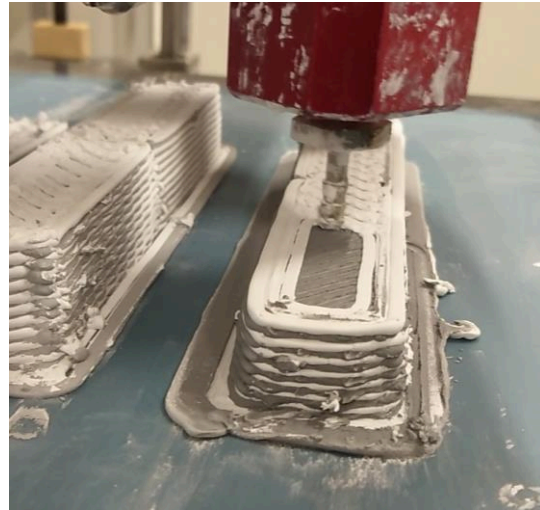
- **Shear thinning behaviour** and **thixotropic** behaviour of the paste
- Reduction in viscosity over the ageing time of the paste → reduced stress for printing



Experimental route



Paste preparation



Material extrusion

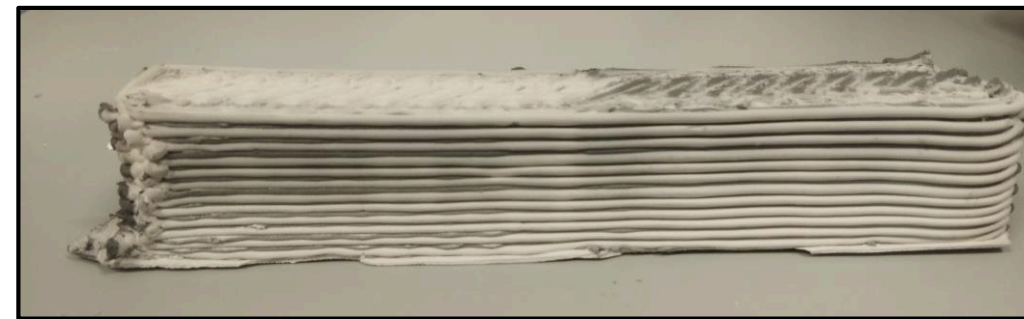
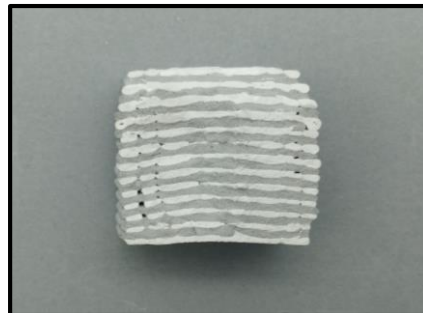


Printed composite bar

- 1) Drying
1) RT --1 K/min-- 60°C; --1 K/min--120°C, free cooling.
- 2) Binder removal
2) RT--5 K/min--180°C; --0.1 K/min--300°C, free cooling.
- 3) Sintering process
3) RT--10 K/min--1100°C; -- 5 K/min--1600°C hold time -2 h; free cooling. (Under Ar atmosphere)



Crushing process

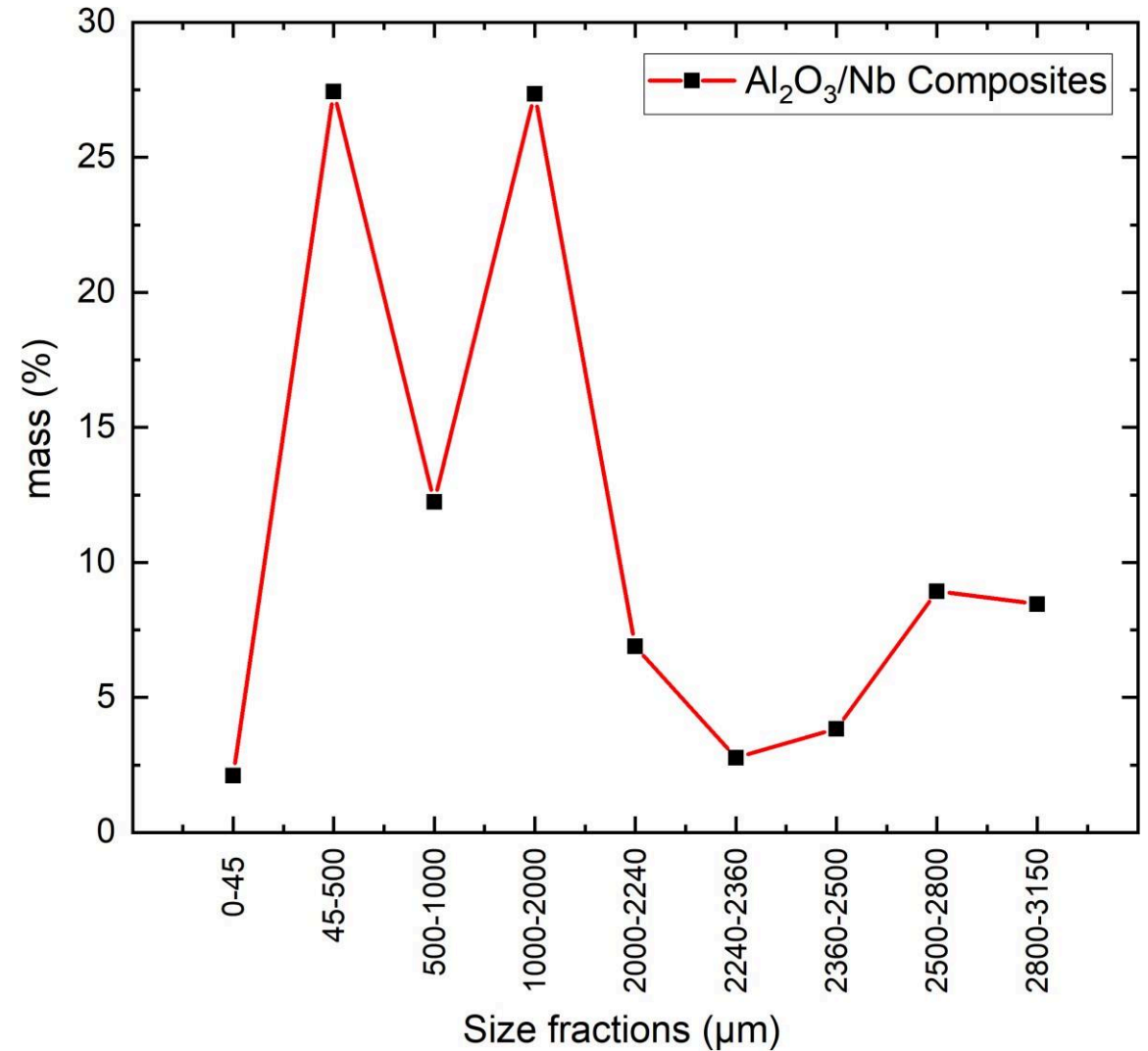


Sintered composite bar

Particle size distribution

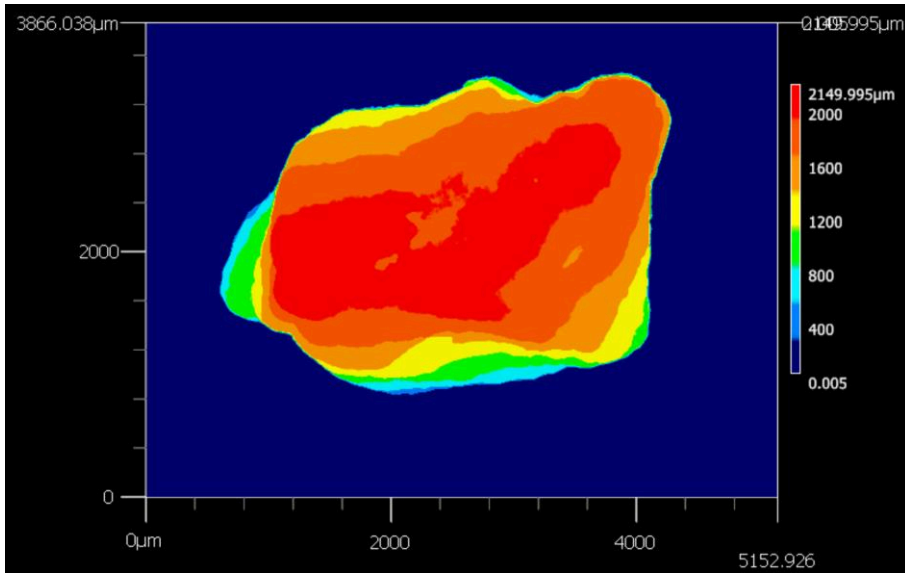


Sieve size (μm)	mass (%)
0-45	2.10
45-500	27.43
500-1000	12.24
1000-2000	27.34
2000-2240	6.89
2240-2360	2.77
2360-2500	3.84
2500-2800	8.93
2800-3150	8.45



Particle morphology

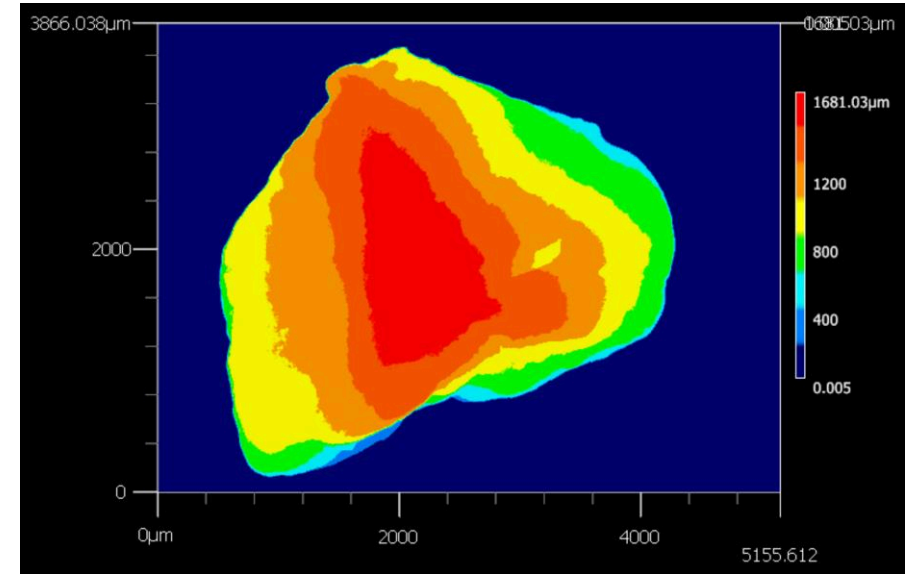
(>75 particles for all aggregates except 0-45 and 45-500 μm)



(2500-2800 μm)

Two types of morphologies:

- **Blocky shape** with a relatively flat plateau
- **Sub-angular blocky** particles with step-wise increment
- better interlocking within the particles



(2240-2360 μm)

- The height of the particle small compared to the width and length of the particle
- **Sharp interface and bonding** between the layers
- Proper distribution of Al_2O_3 -Nb materials in all aggregate classes



500-1000 μm



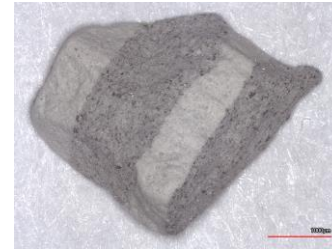
1000-2000 μm



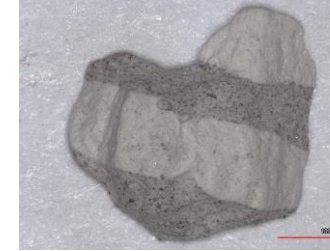
2000-2240 μm



2240-2360 μm



2360-2500 μm

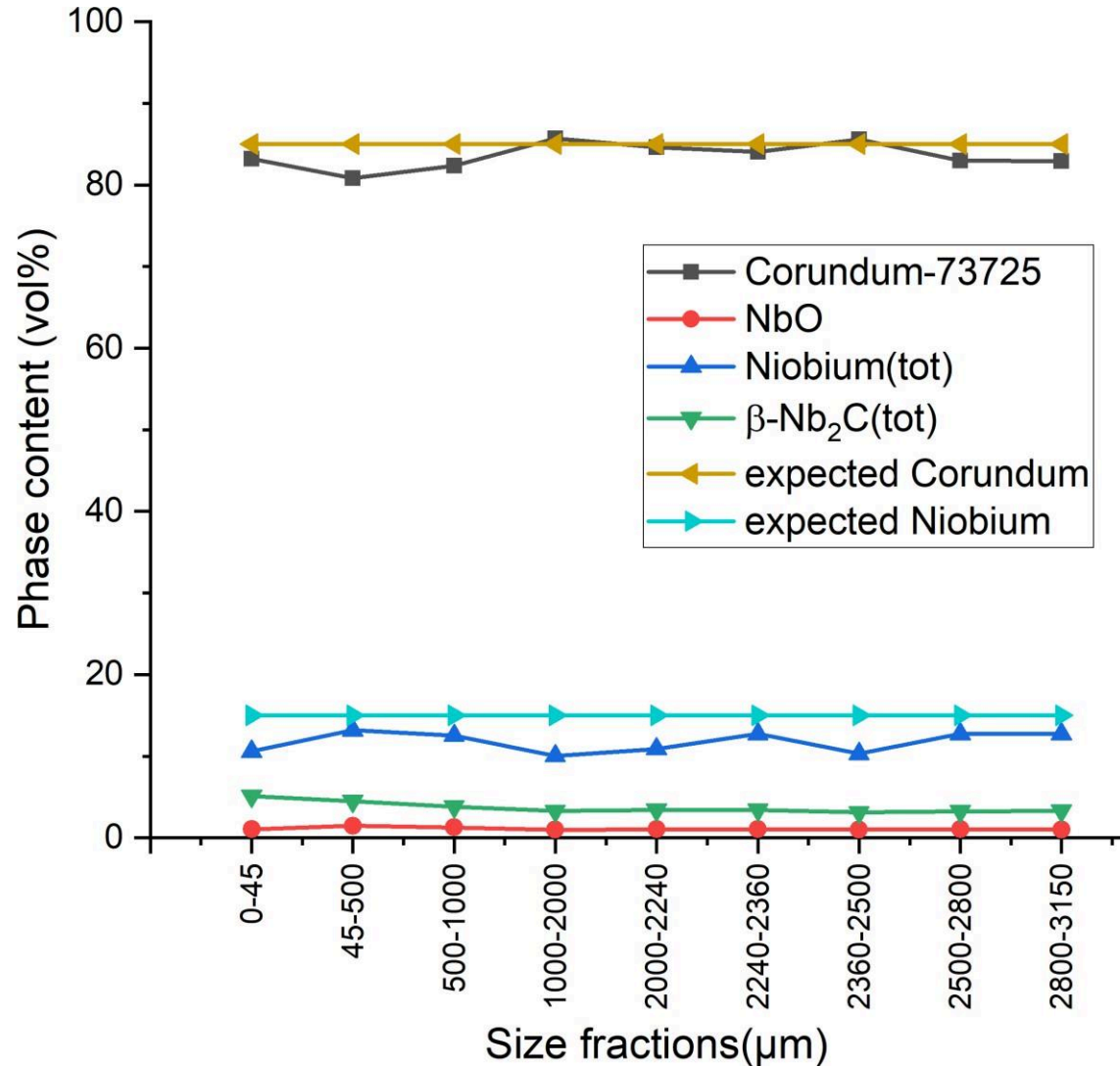


2500-2800 μm



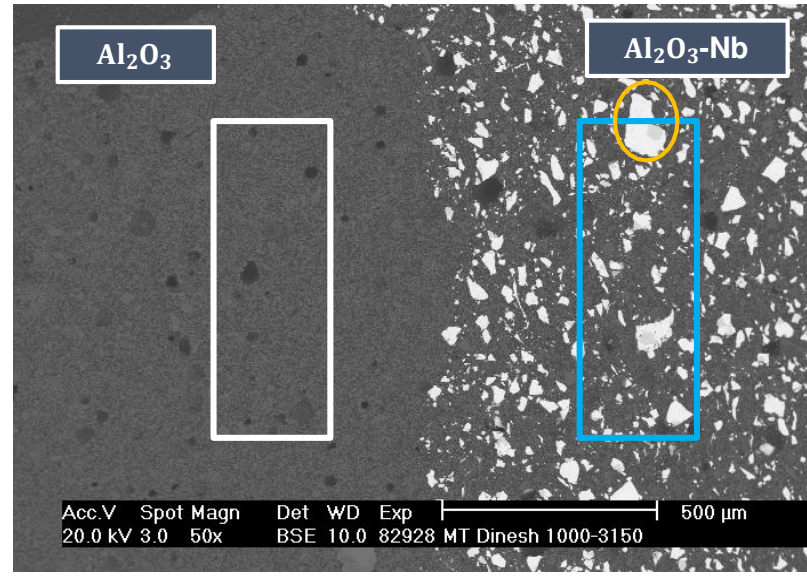
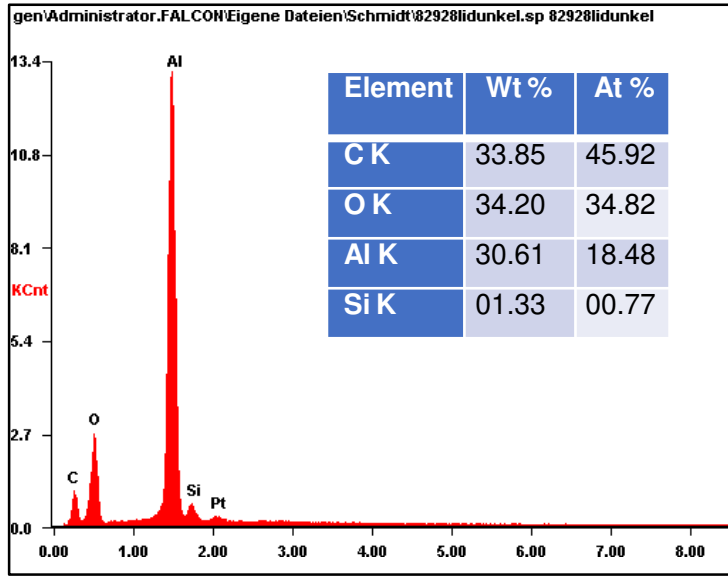
2800-3150 μm

XRD analysis

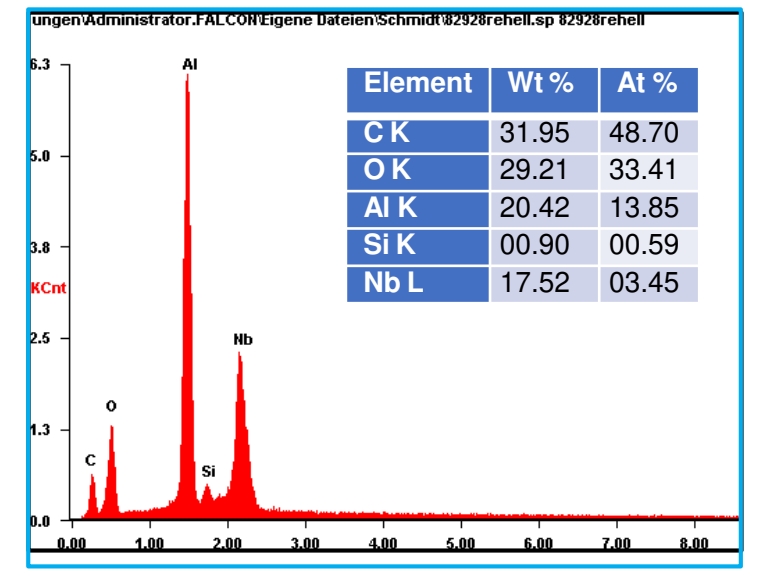


- Homogeneous distribution of phase, nearly reaching the expected nominal composition of the material (85 vol% corundum & 15 vol% of niobium)
- NbO: 1-1.5 vol%
- β-Nb₂C: 3-5 vol%
- **Presence of NbO:** Chemical interactions with environment, sintering process, impurity from initial powders
- **Presence of β-Nb₂C:** CO /CO₂ from the graphitic sintering furnace, diffusion of such gases through porosity

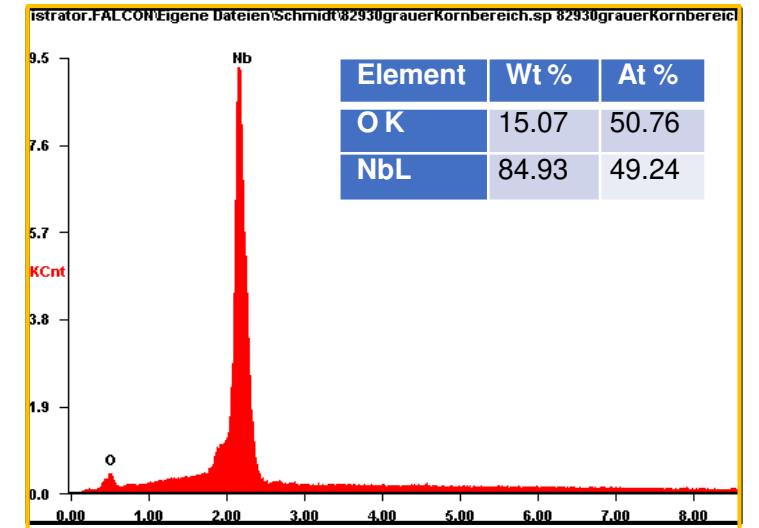
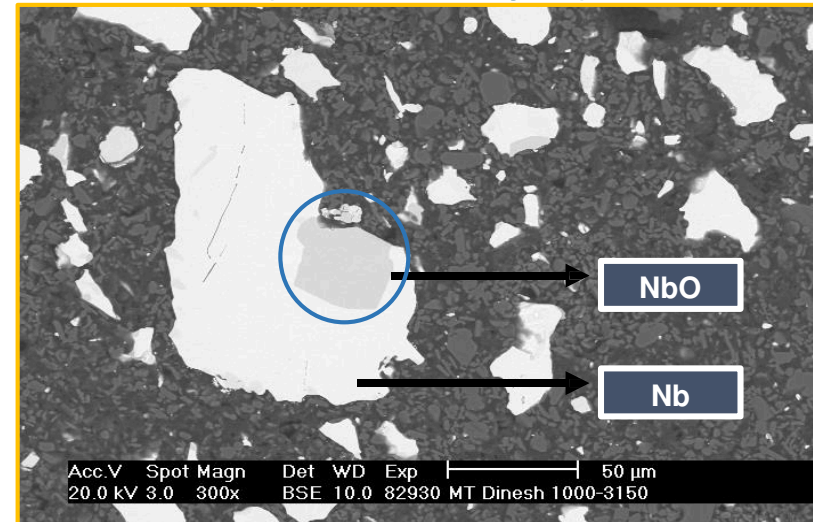
SEM and EDX analysis



(2800-3150 µm)



- Clear and distinct interface layers of Al₂O₃ and Al₂O₃ - Nb
- Presence of C attributed to resin from graphitic furnace, residual organics
- Presence of Niobium oxide approved by elemental distribution

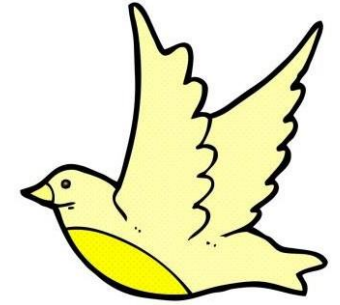
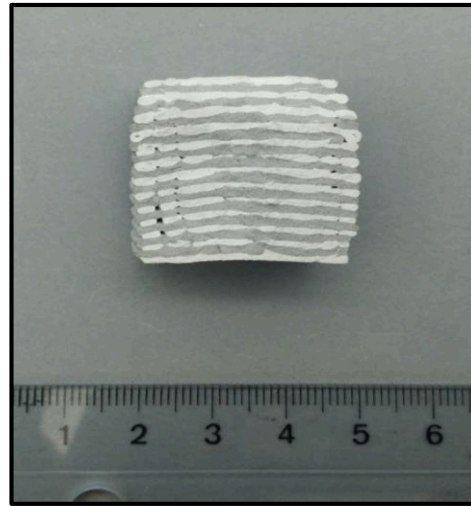
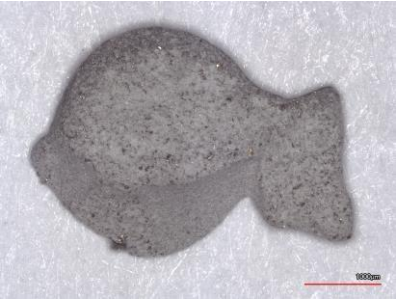


Summary

- Successful production of **Al₂O₃ & Nb refractory composites** using novel **additive manufacturing technique** based on paste extrusion
- Development of **paste formulation** with required shear thinning behaviour with optimum printing parameters
- Clear and sharp interface between both the **Al₂O₃ & Al₂O₃ -Nb** layers
- **Homogeneous distribution** of the respective **Al₂O₃ & Nb phases** with the nominal verified by XRD analysis and supported by elemental analysis from SEM with presence of the impurity phases NbO and β -Nb₂C
- **No diffusion of Nb** into the alumina grains, ensuring the chemical stability during application

Futuristic scope

- Proper **optimisation of the paste and printing parameters** with good quality printing
- Production of composites with these pre-synthesised granules and characterisation of **mechanical and thermal properties of those refractory composites**



Thank you all for your attention!



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