

End of semester IV Presentation

# Preparation and characterization of Oxide Dispersion Strengthened (ODS) steels.

Presented by:

**BEN ZINE** Haroune Rachid

Under the supervision of:

**Dr. Balàzsi** Csaba

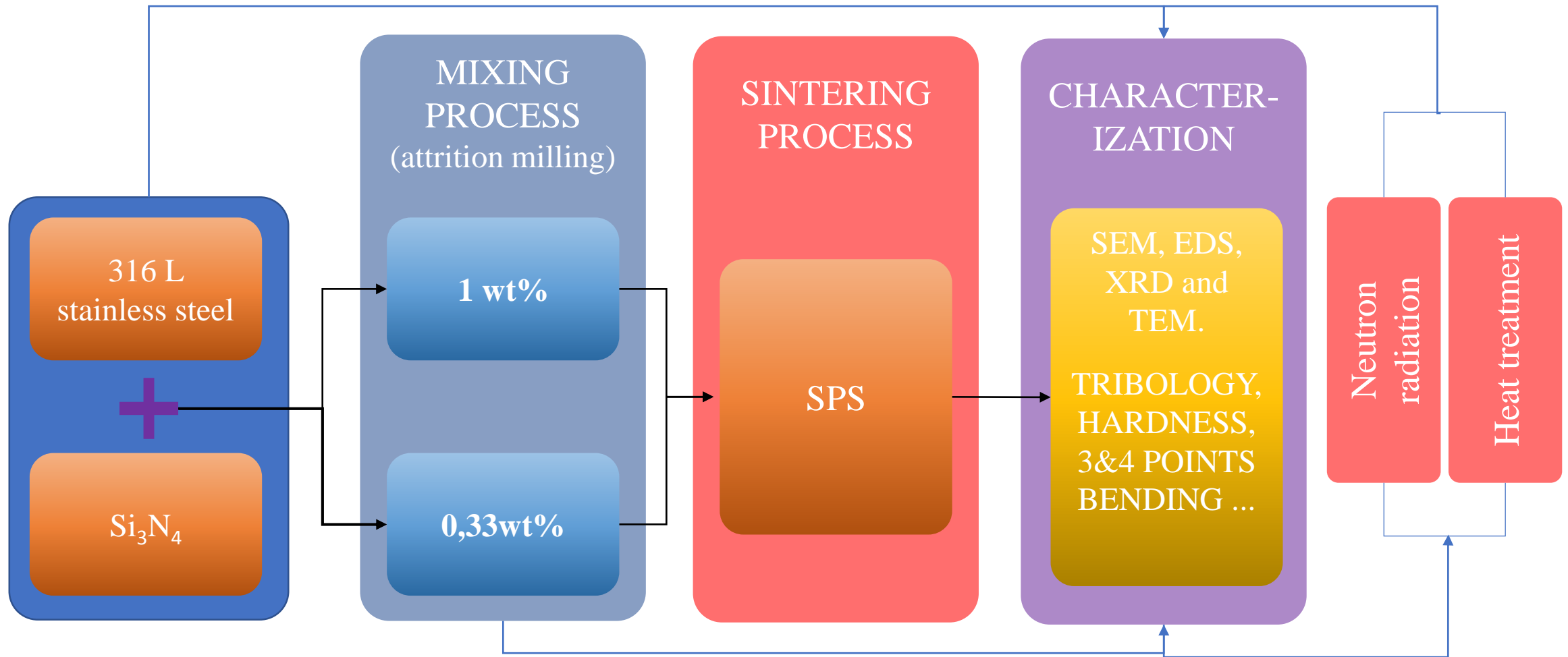
**Dr. Balàzsi** Katalin

# Content:



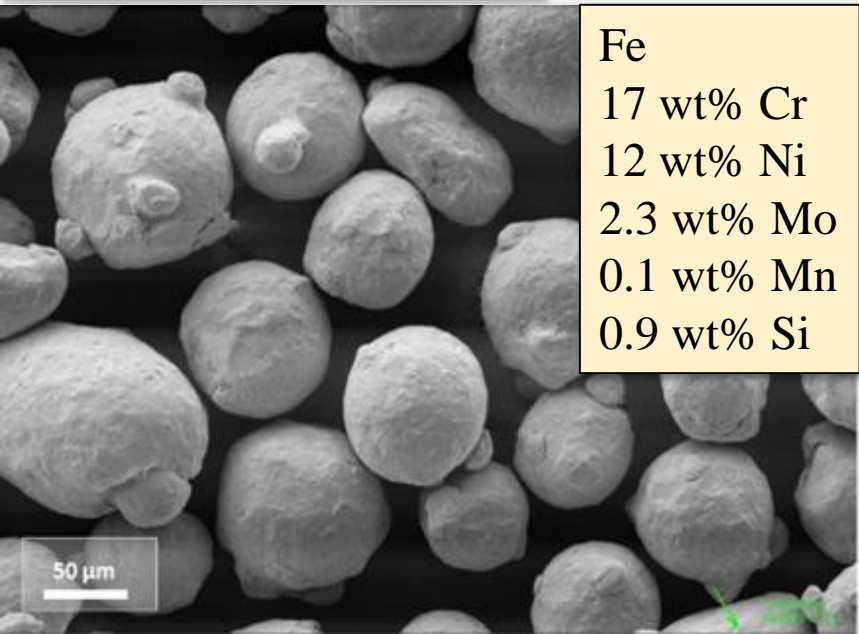
- Experiments work Plan
- First Experimental results:
  - 316L Höganäs+ 0.33wt% Si<sub>3</sub>N<sub>4</sub>
  - 316L Höganäs+ 1wt% Si<sub>3</sub>N<sub>4</sub>
- Results Comparison
- Conclusion
- Results of the actual semester
- Future work plans

# Experiments Work Plan:

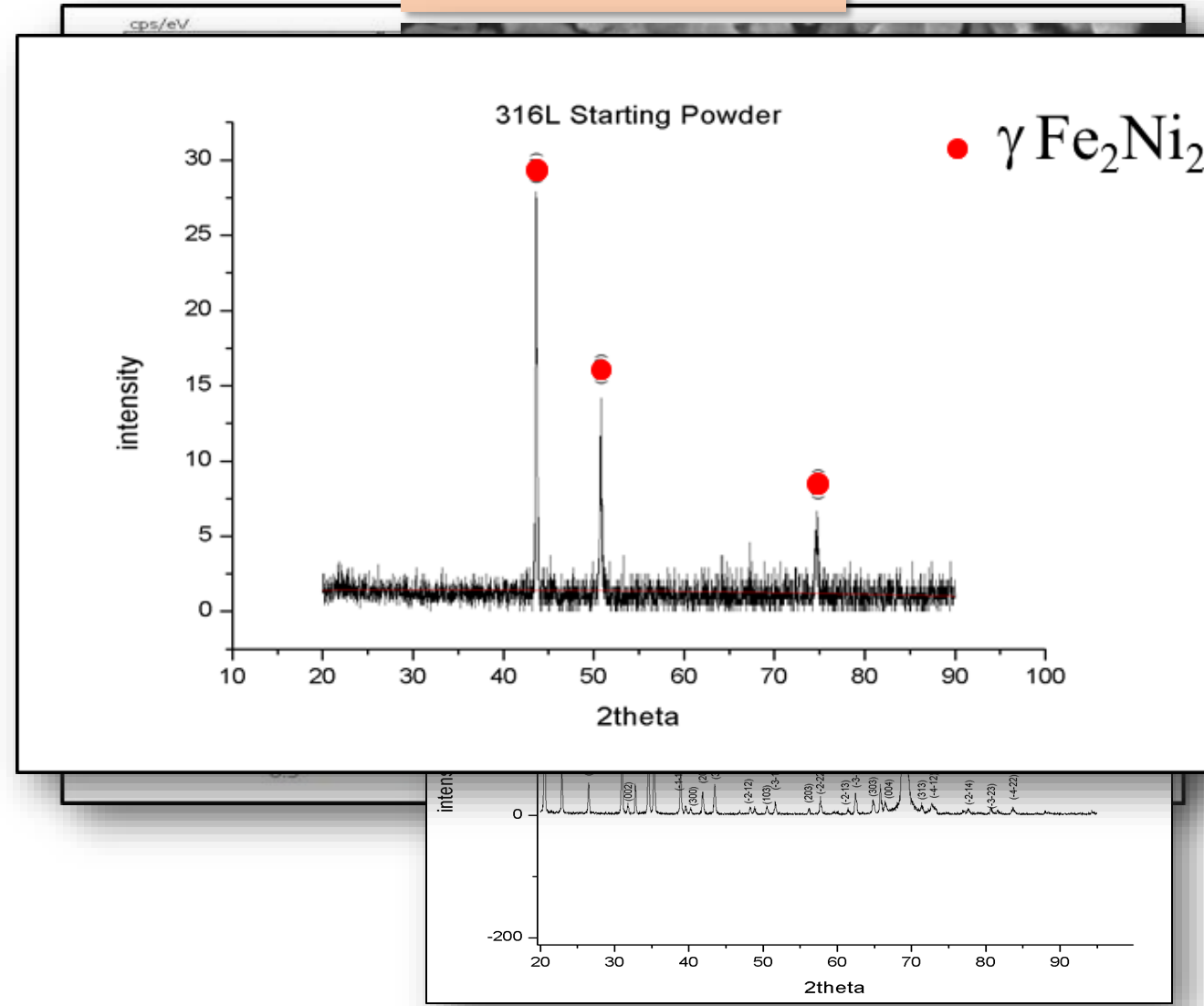


# Structure of based materials

316L Höganäs (reference)

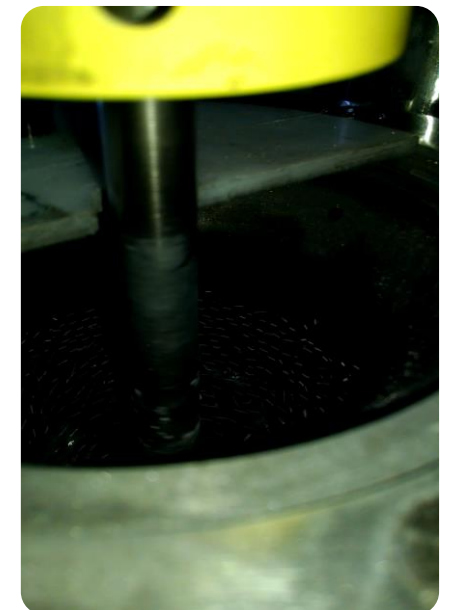


UBE Si<sub>3</sub>N<sub>4</sub> (addition)



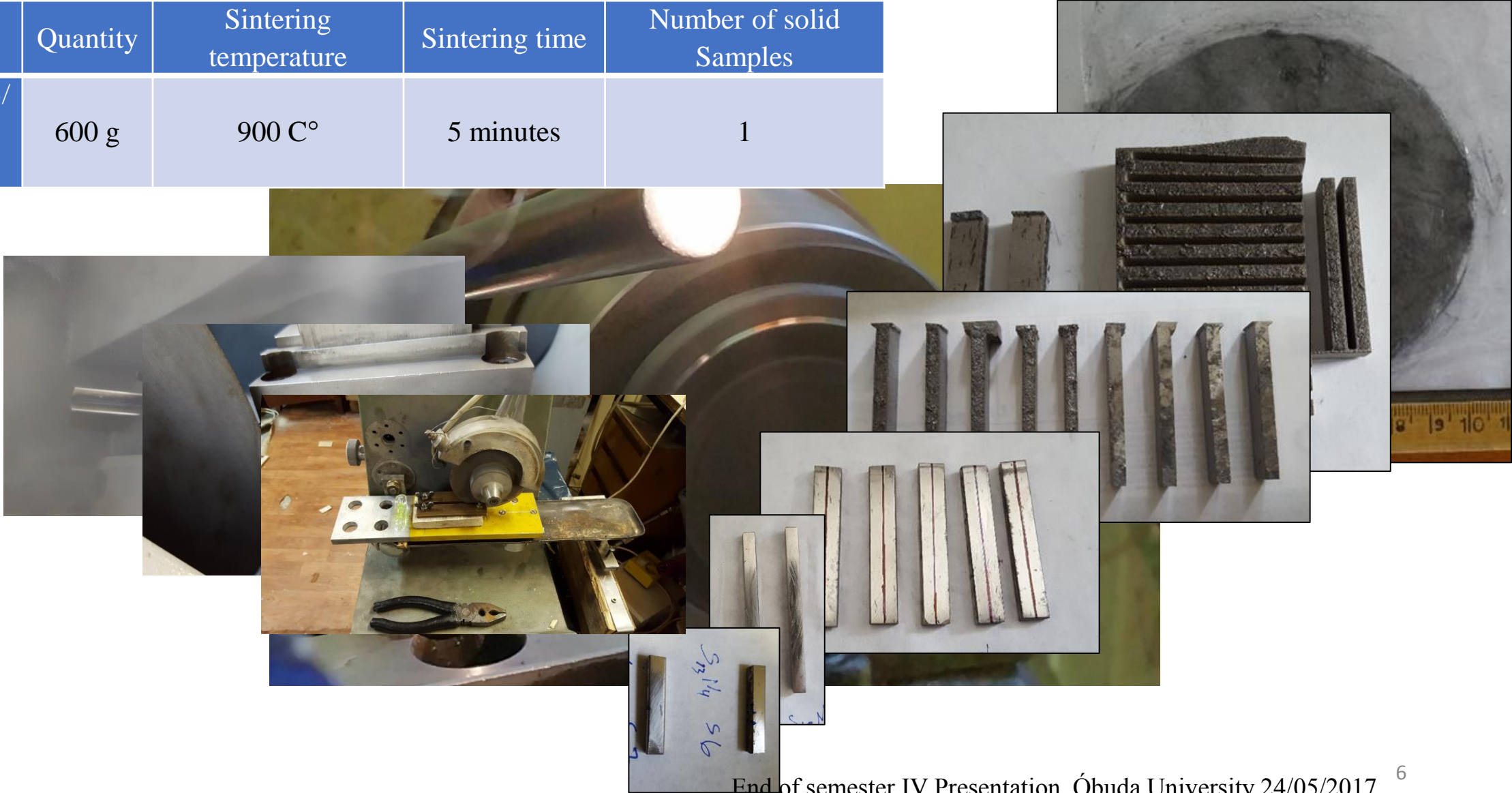
# High efficient attrition milling:

Material	Mass	Grinding material		Container	Milling type		Milling Speed	Milling time	Drying conditions	
		Diameter	Quantity		Wet	Quantity			Spinning speed	Drying temperature
Höganäs 316 L (SS)	300g	Stainless steel Balls		Big	Wet	Quantity	603 rpm	5 hours		
Si <sub>3</sub> N <sub>4</sub>	3g	3 mm	2.9 kg	1400 ml	ethanol	300 ml			100 rpm	75 C°



# Sintering Process: Spark Plasma Sintering (SPS):

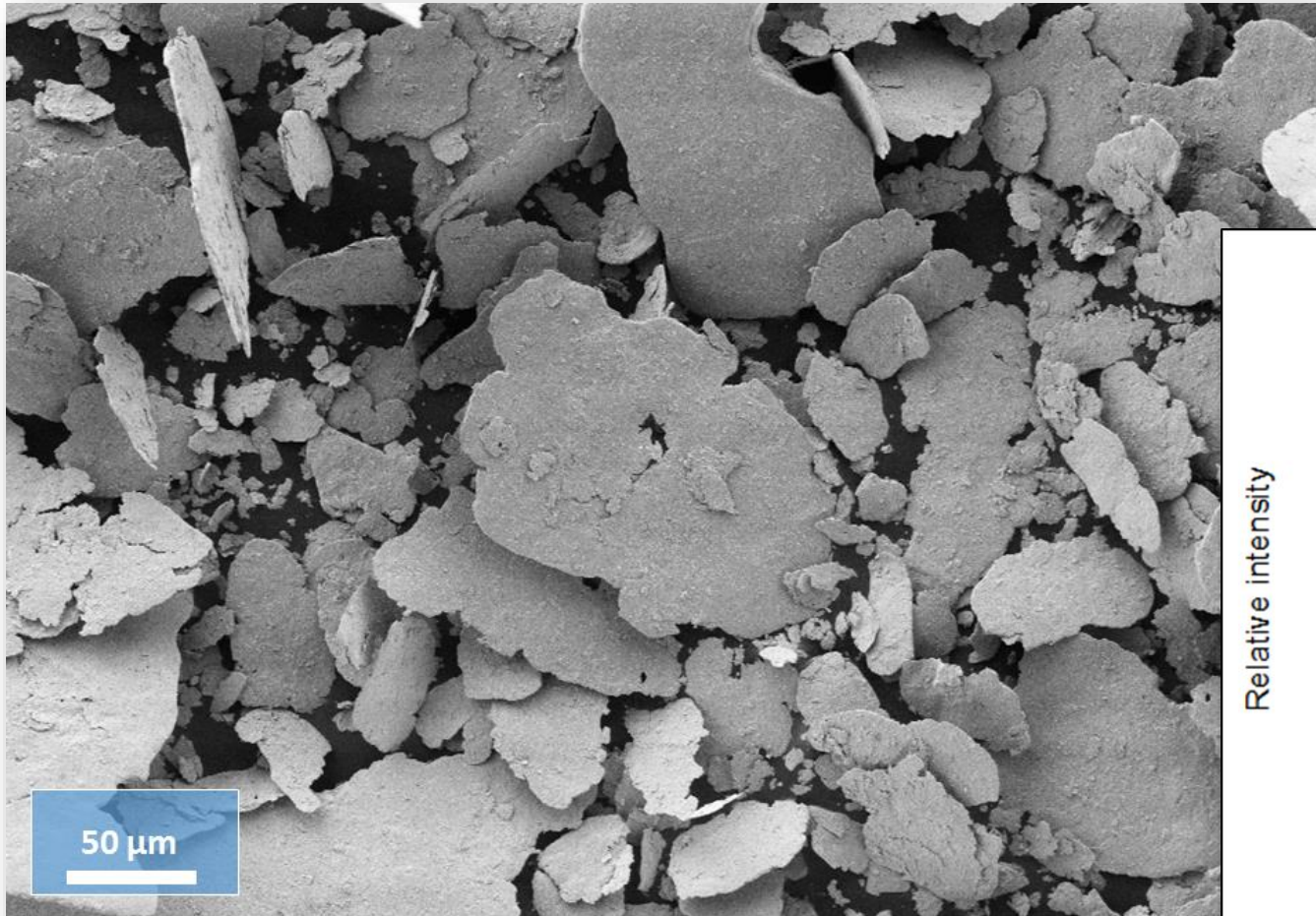
Alloy	Quantity	Sintering temperature	Sintering time	Number of solid Samples
316L+0.33/ 1 wt% Si3N4	600 g	900 C°	5 minutes	1



316L Hőganäs + 0.33 wt%  
 $\text{Si}_3\text{N}_4$

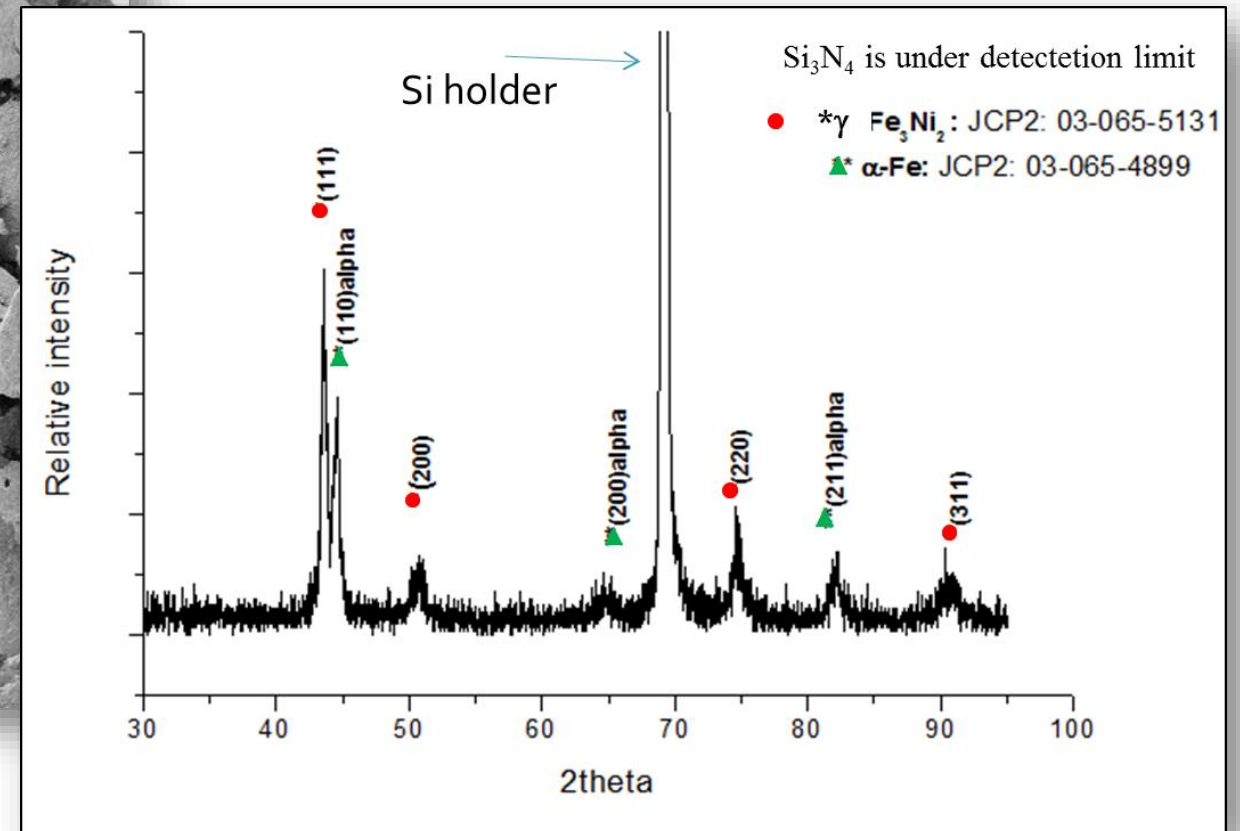


# Structure of milled 316L / 0.33 wt% Si<sub>3</sub>N<sub>4</sub>



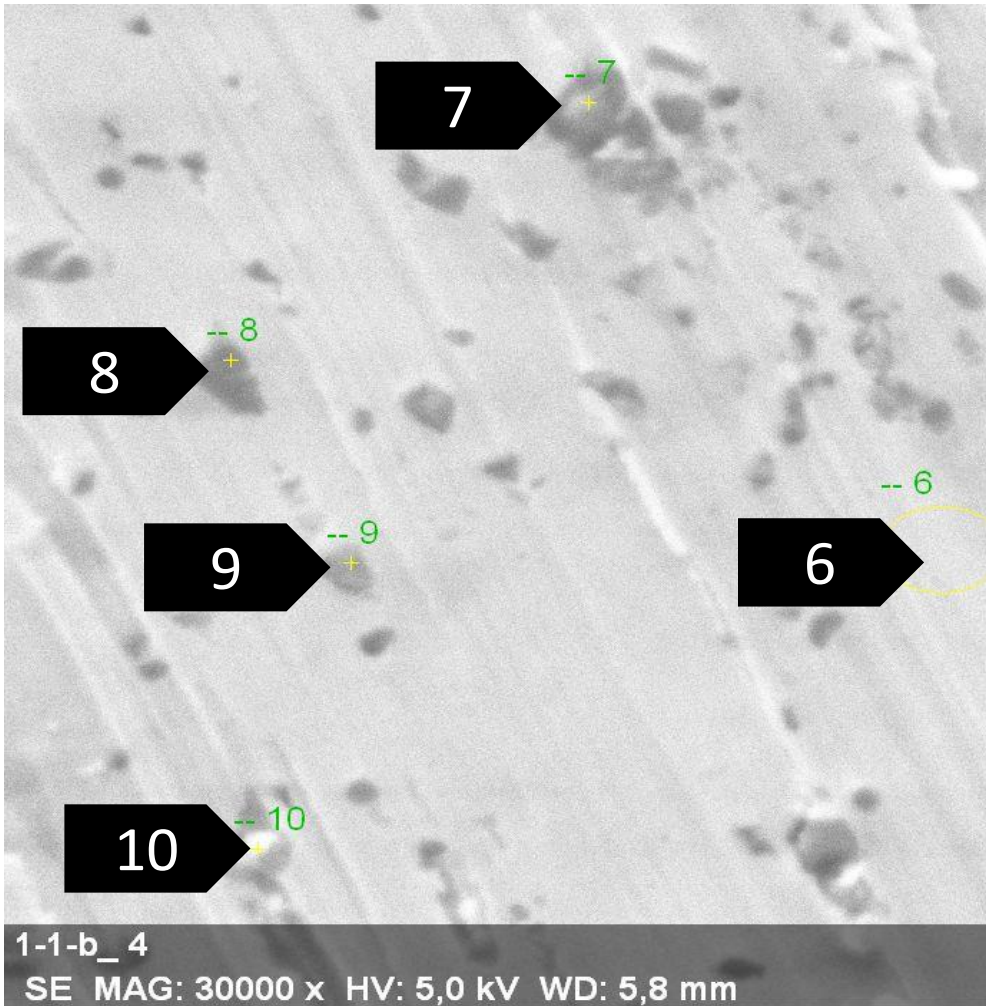
SEM image of milled powder with lamellar grains

XRD spectrum of milled powder

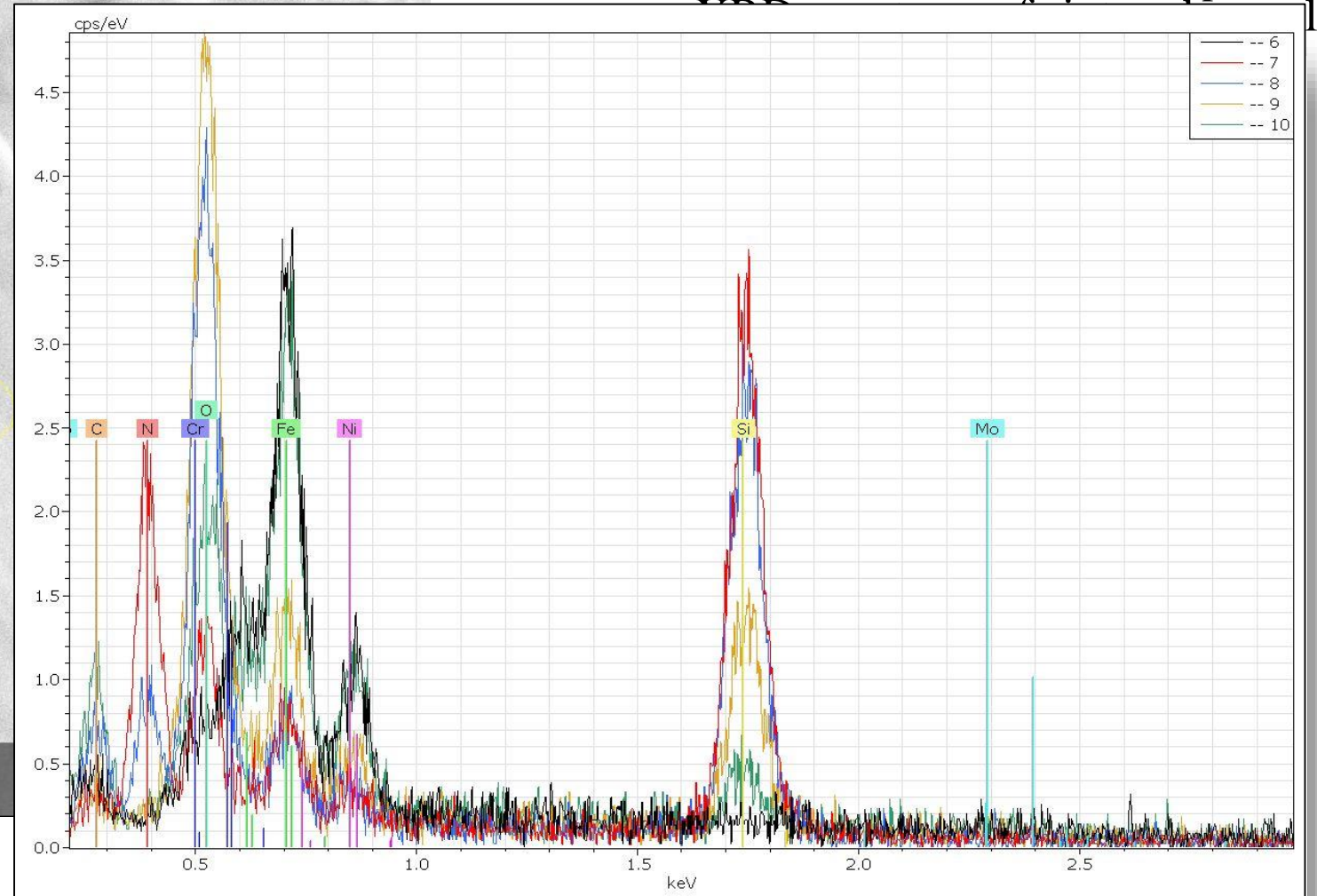




# Structure of sintered 316L / 0.33 wt% Si<sub>3</sub>N<sub>4</sub>

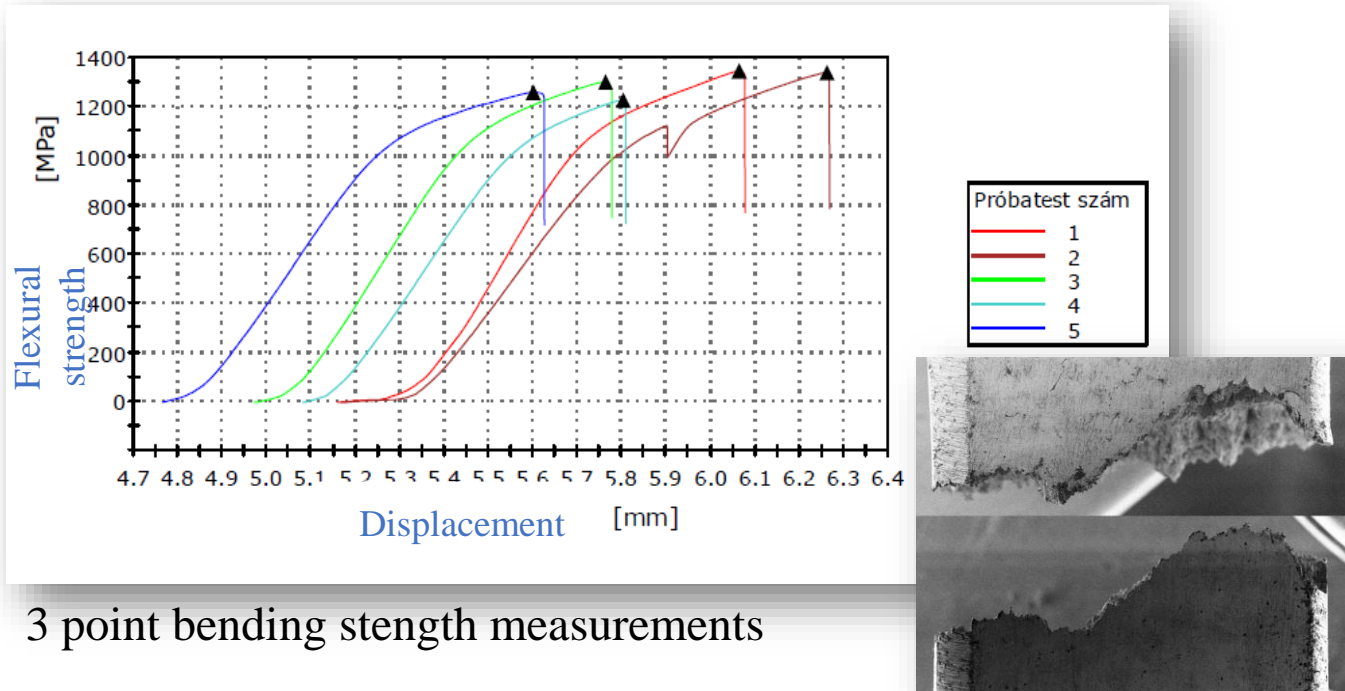


EDS spectra of the 316L+ 0.33 wt % Si<sub>3</sub>N<sub>4</sub> Sintered Sample



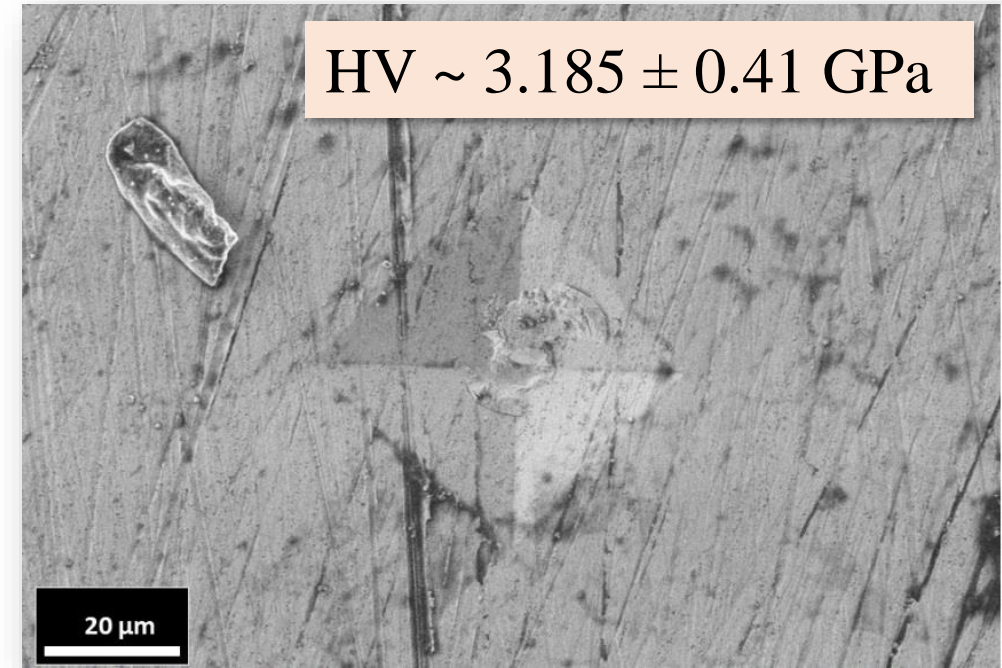
# Properties of sintered 316L / 0.33 wt% Si<sub>3</sub>N<sub>4</sub>

SEM image of indentation



3 point bending strength measurements

3p bending strength ~ 1472 MPa (\*5% error)



SEM image of sample after tribology test

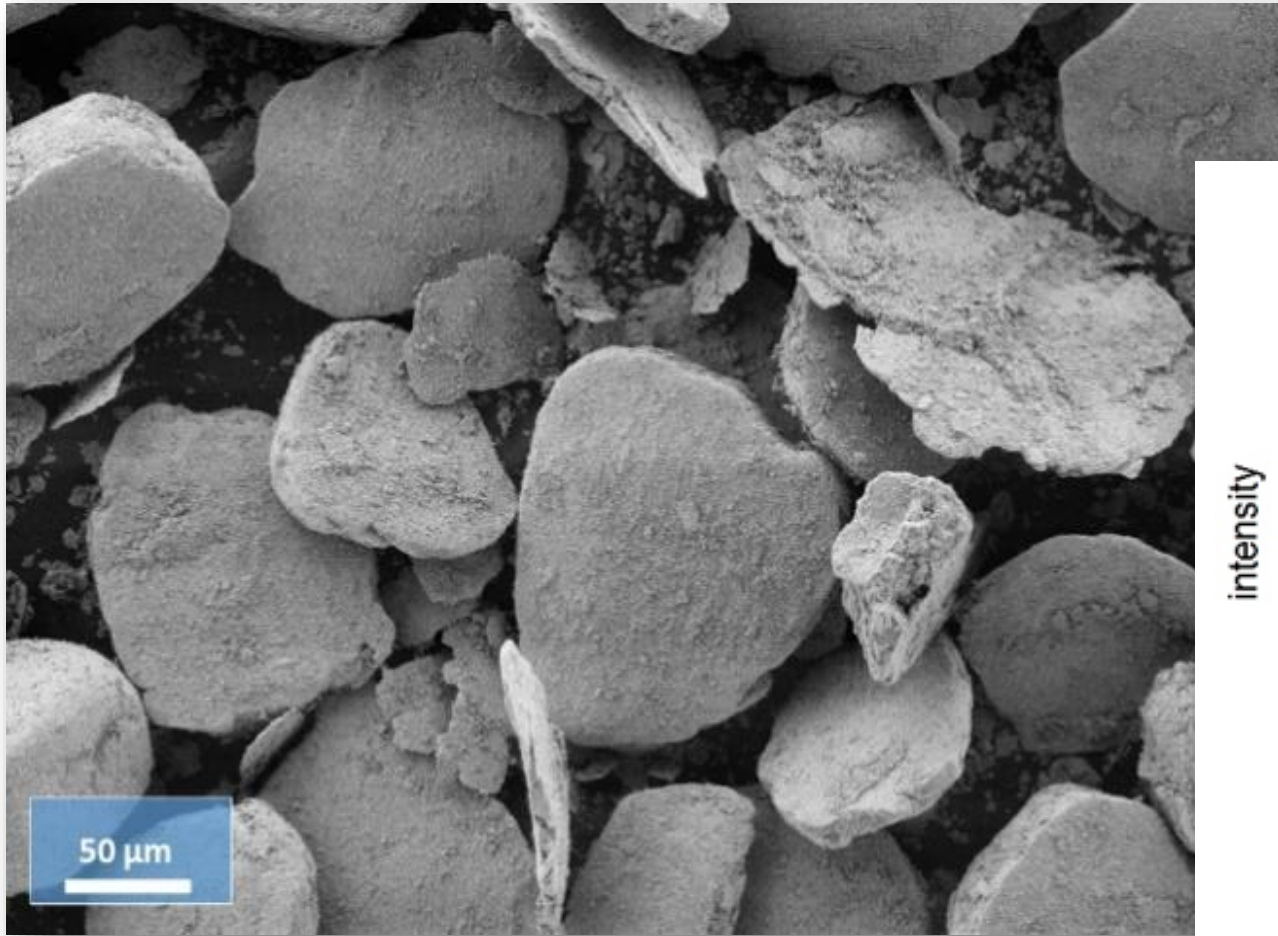


Friction coefficient  $\mu \sim 0.8$

316L Hőganás + 1 wt% Si<sub>3</sub>N<sub>4</sub>

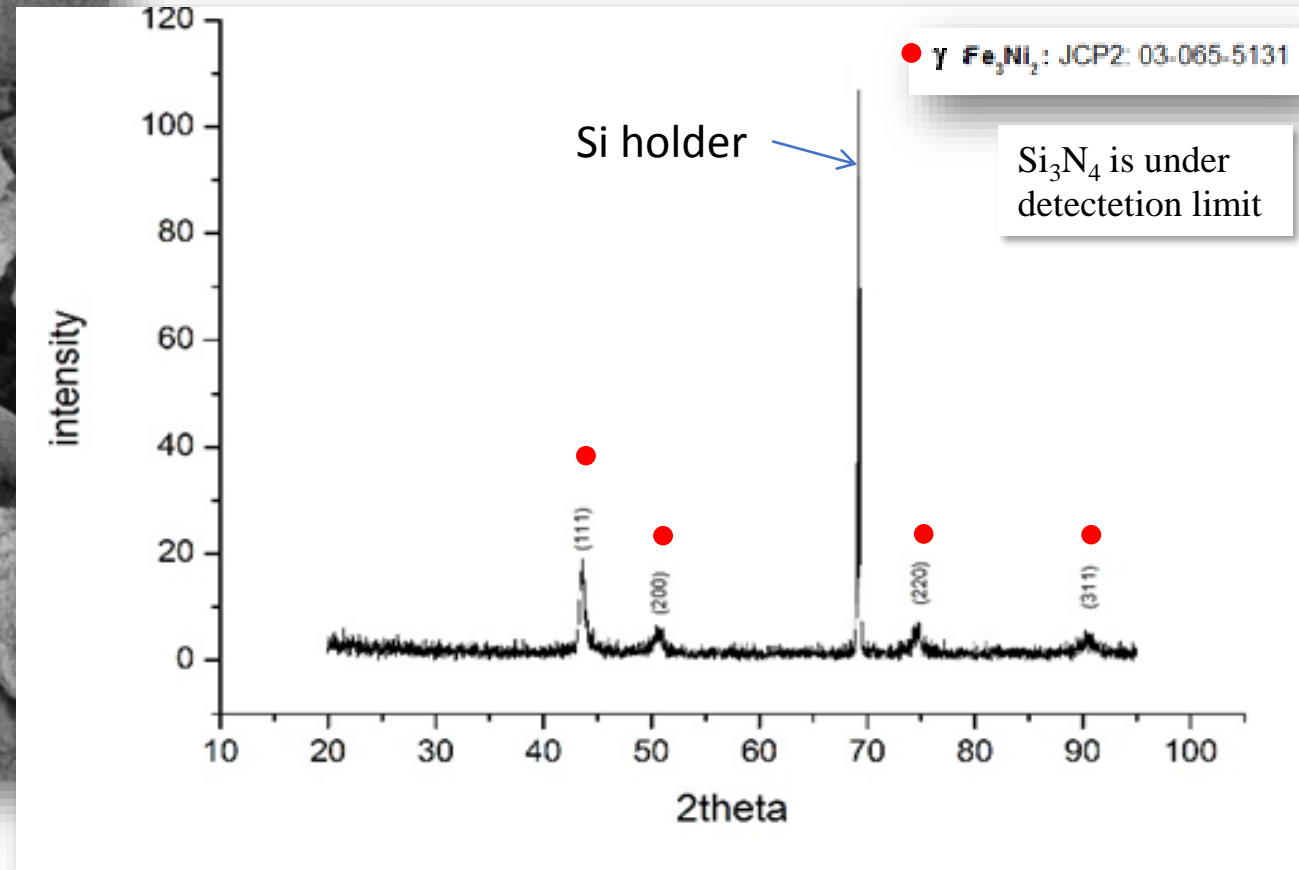


# Structure of milled 316L / 1 wt% Si<sub>3</sub>N<sub>4</sub>

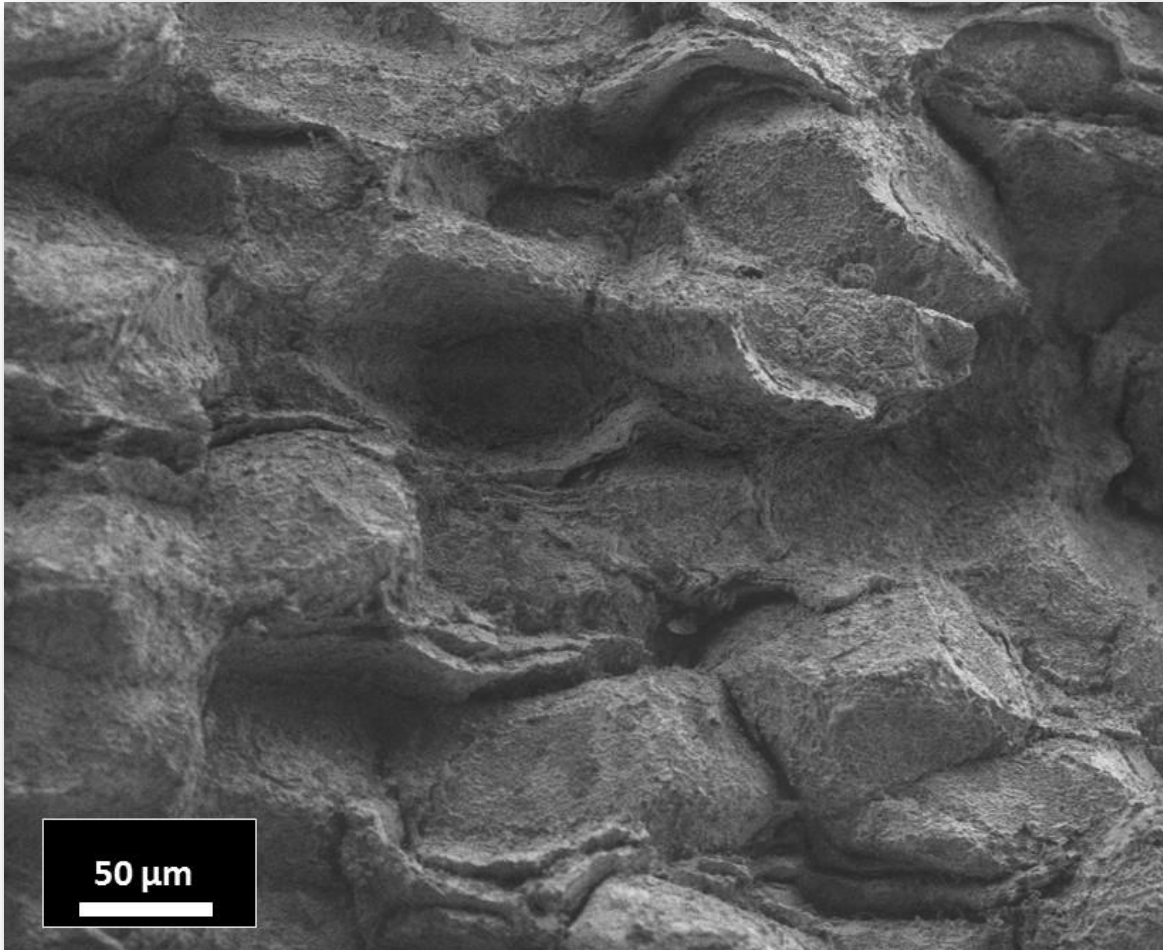


SEM image of milled powder with lamellar grains

XRD spectrum of milled powder

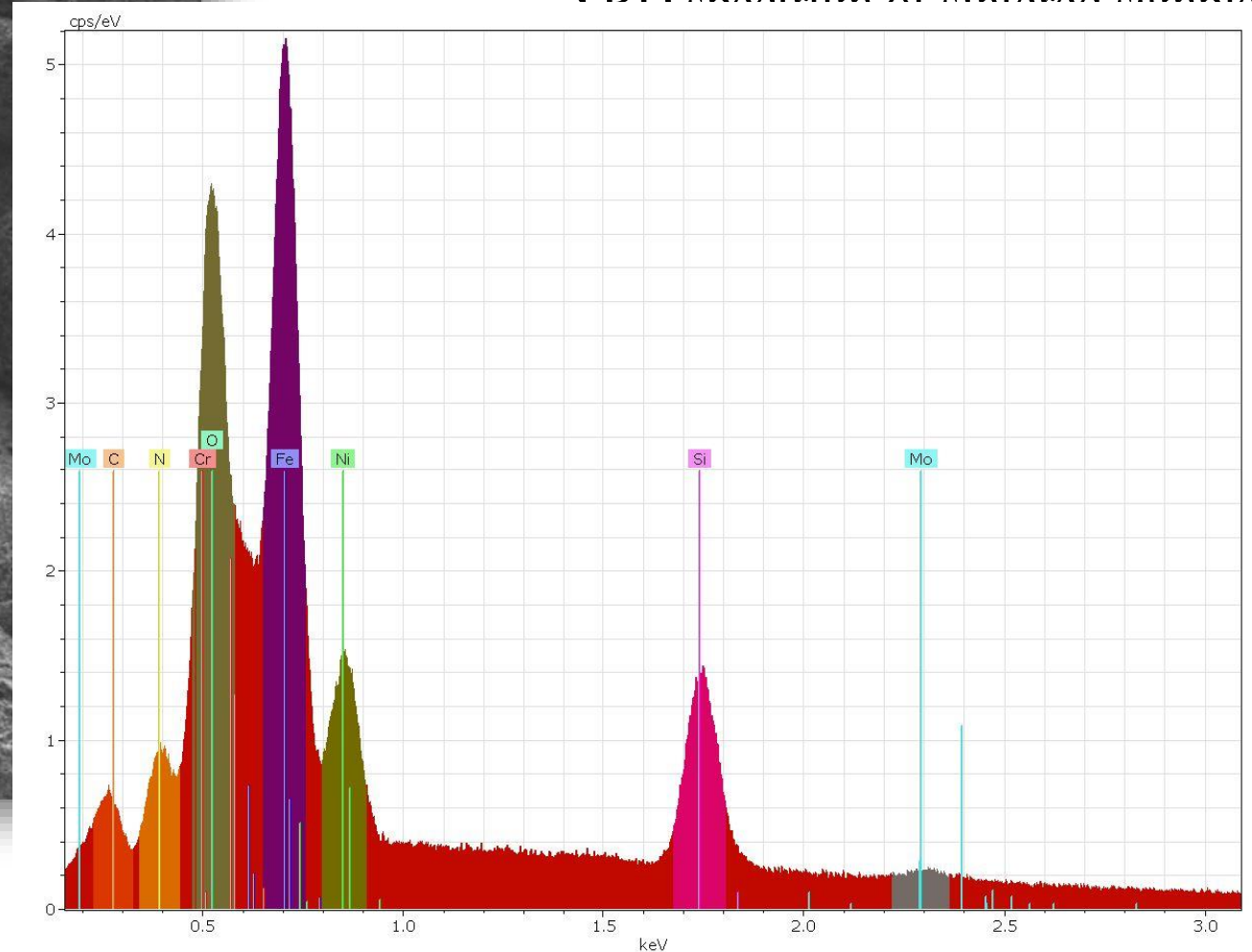


# Structure of sintered 316L / 1 wt% Si<sub>3</sub>N<sub>4</sub>

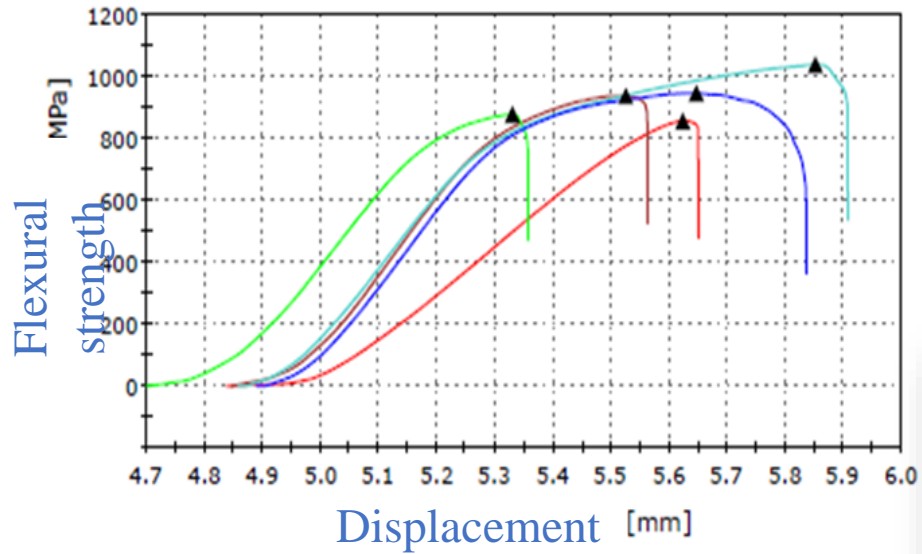


SEM image of fracture surface of sintered sample

EDS spectra of the 316L+ 1wt %Si<sub>3</sub>N<sub>4</sub> Sintered Sample  
EDS spectrum of sintered sample

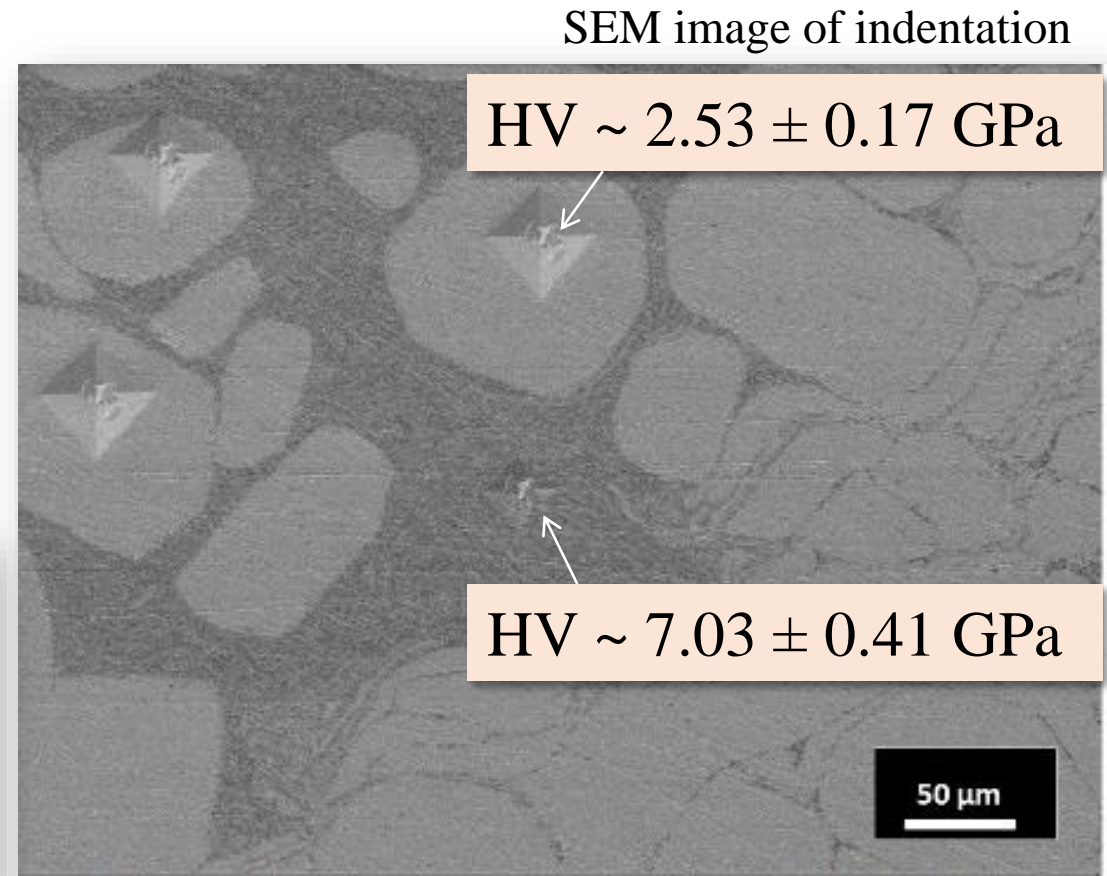
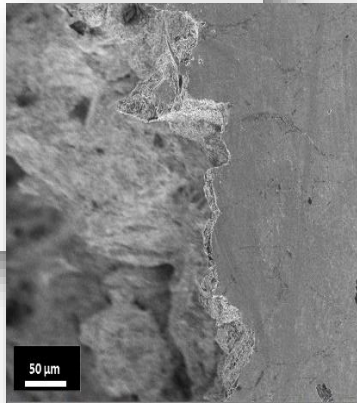


# Properties of 316L / 1 wt% Si<sub>3</sub>N<sub>4</sub>



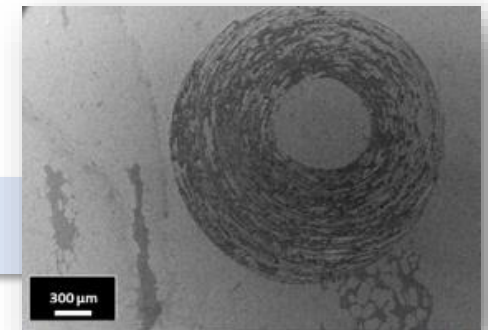
3 point bending strength measurements

3p bending strength ~ 932 MPa (\*5% error)



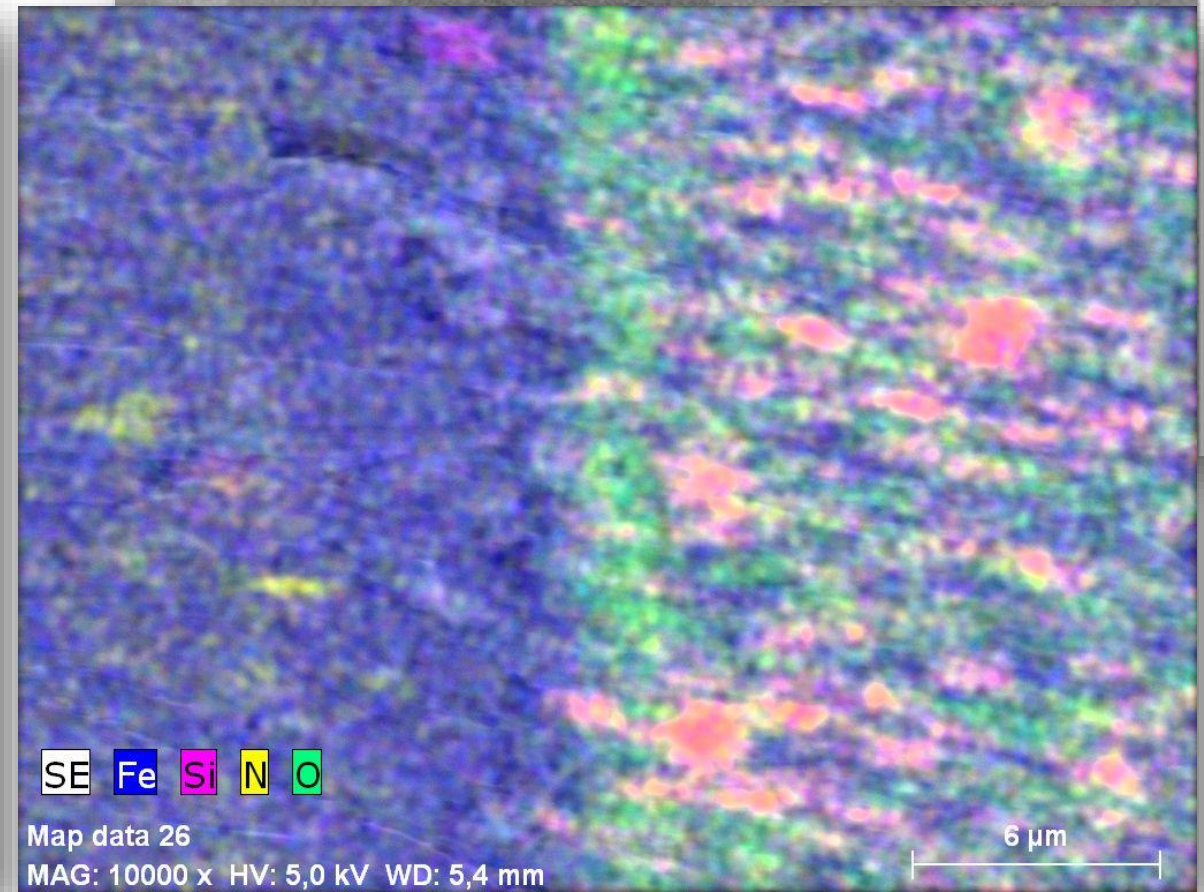
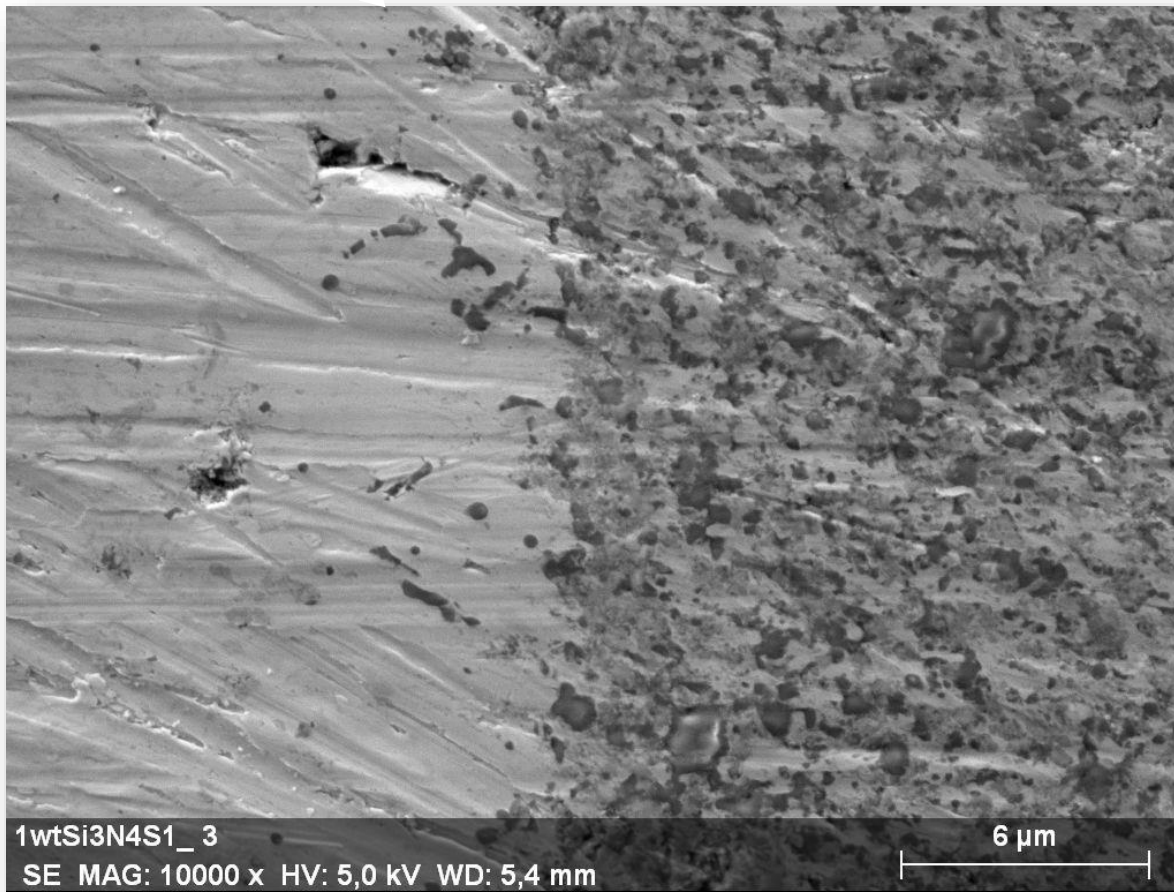
SEM image of sample after tribology test

Friction coefficient  $\mu \sim 0.8$





Elemental map of sintered 316 L / 1 wt%  $\text{Si}_3\text{N}_4$

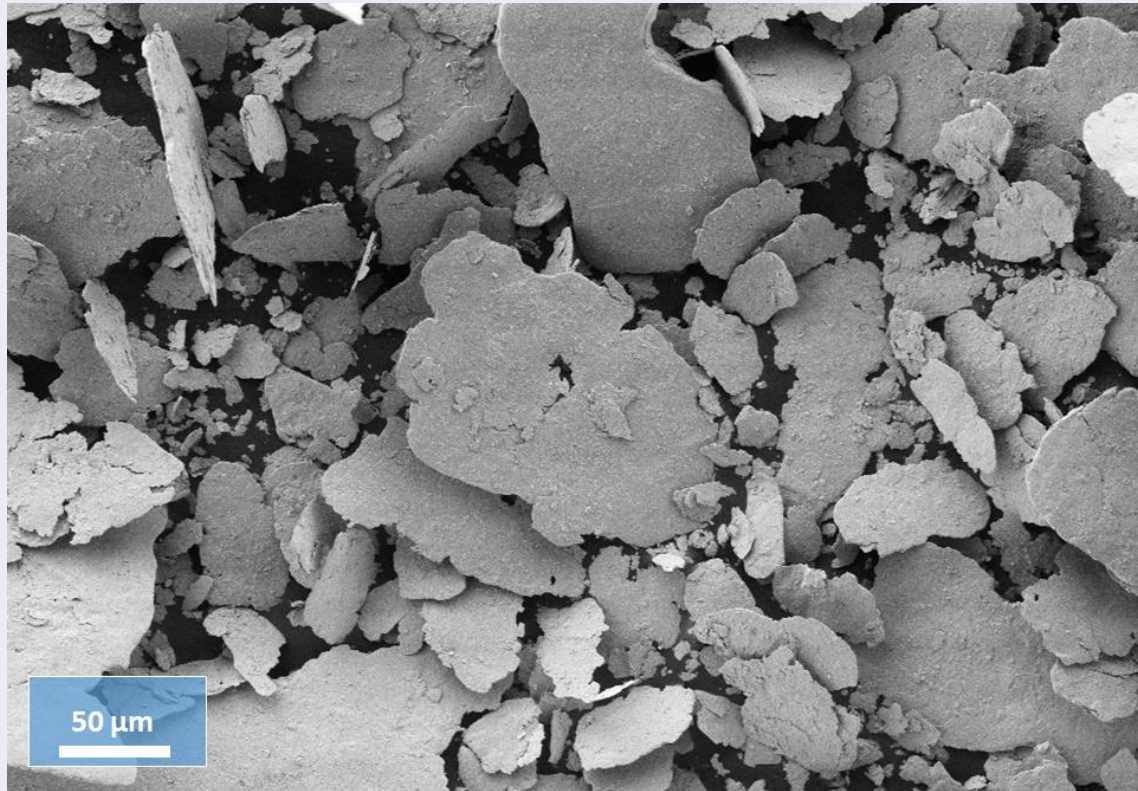


# Results Comparison

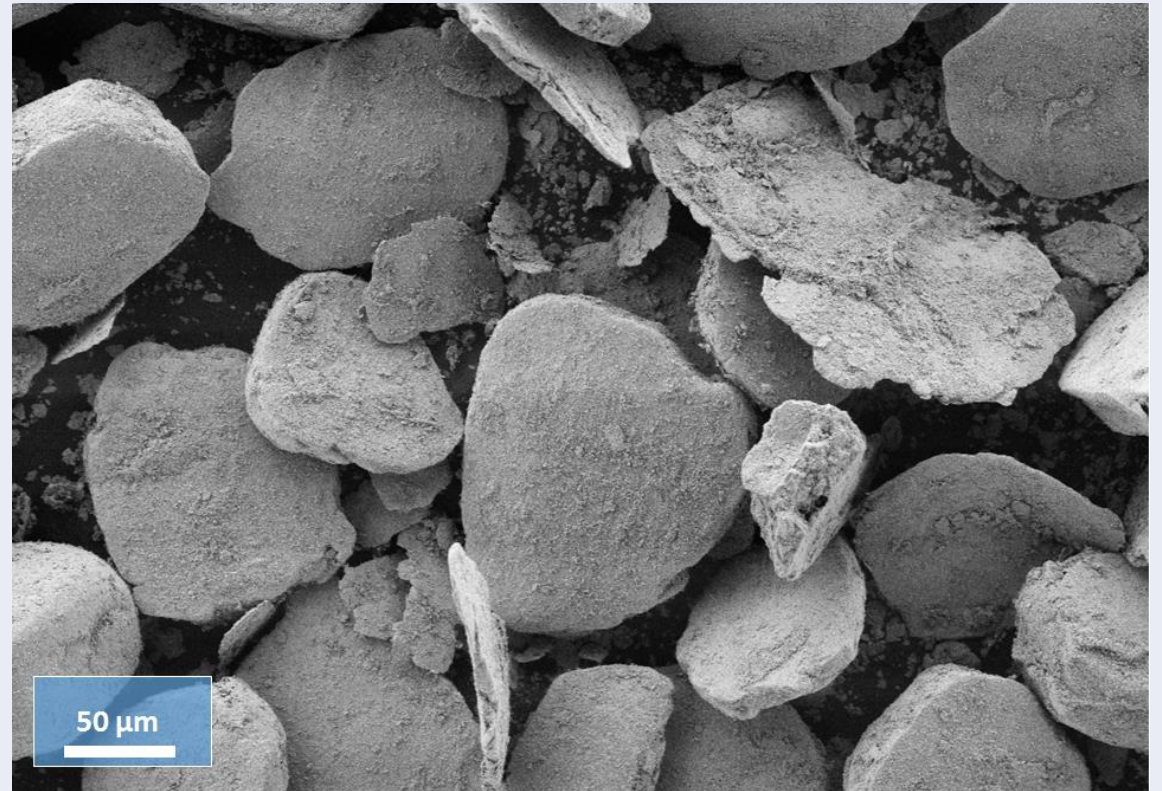


# Morphology after Milling Process

316L Hoganas + **0.33**wt% Si<sub>3</sub>N<sub>4</sub>

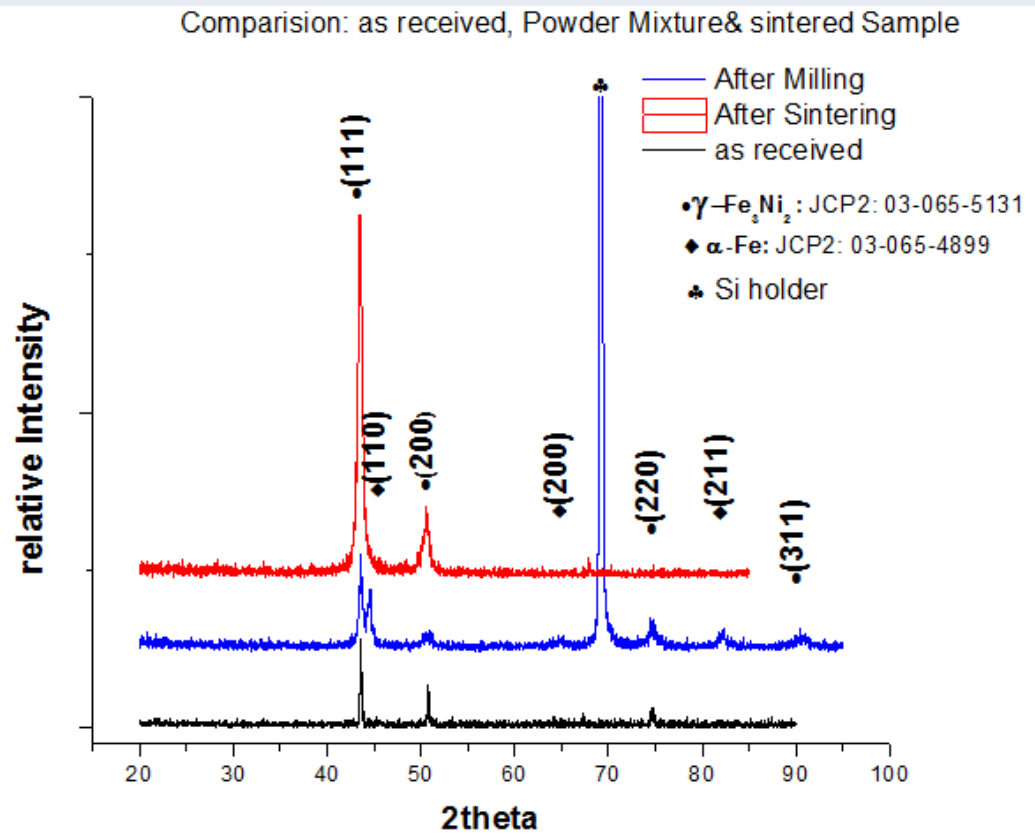


316L Hoganas + **1** wt% Si<sub>3</sub>N<sub>4</sub>

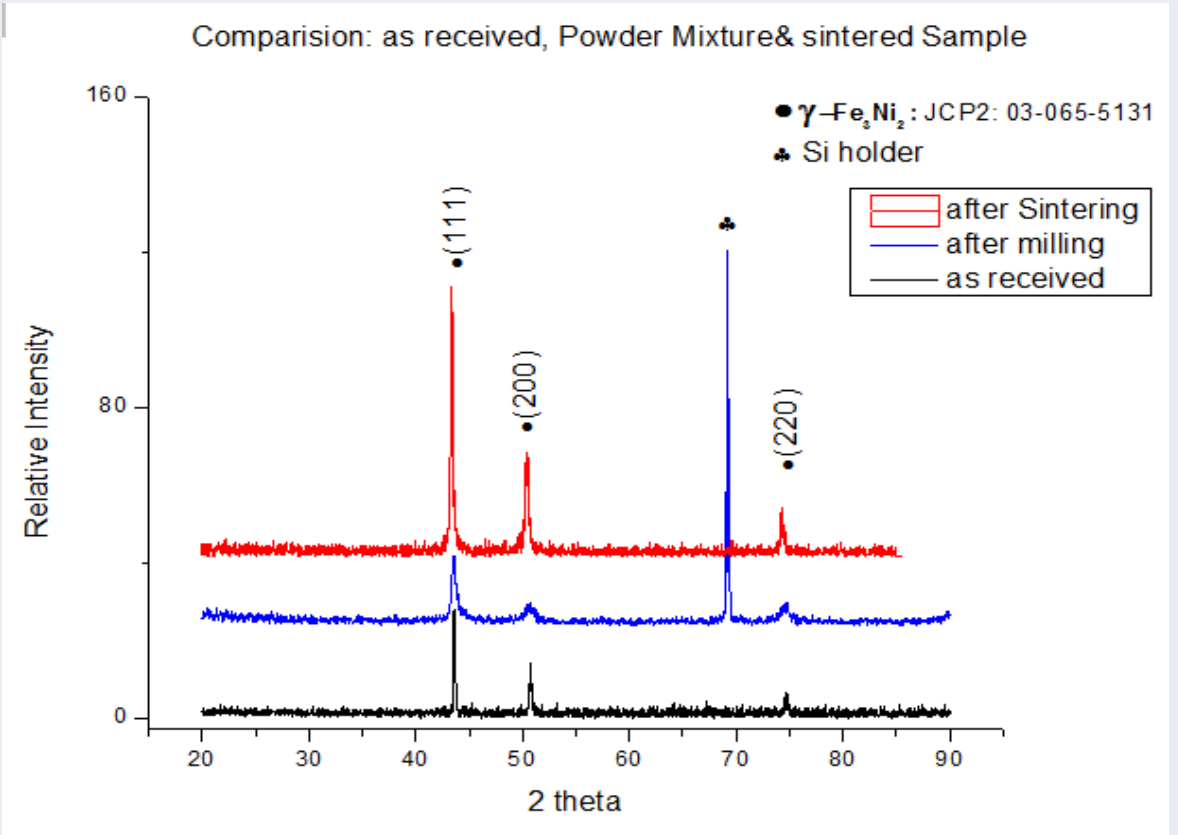


# XRD

316L Hoganas +0.33wt% Si3N4

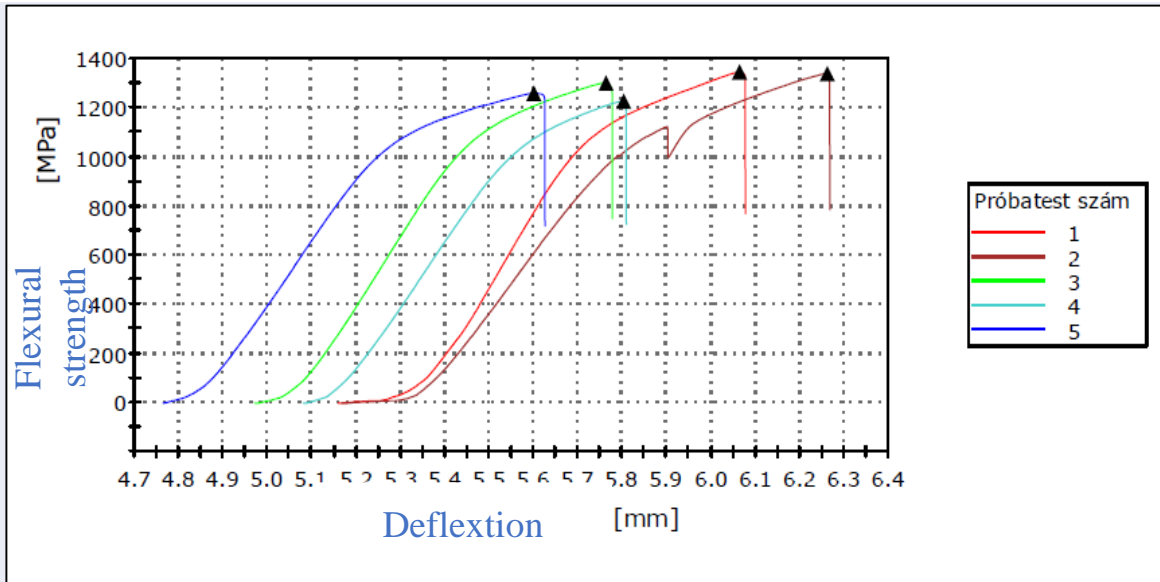


316L Hoganas +1 wt% Si3N4



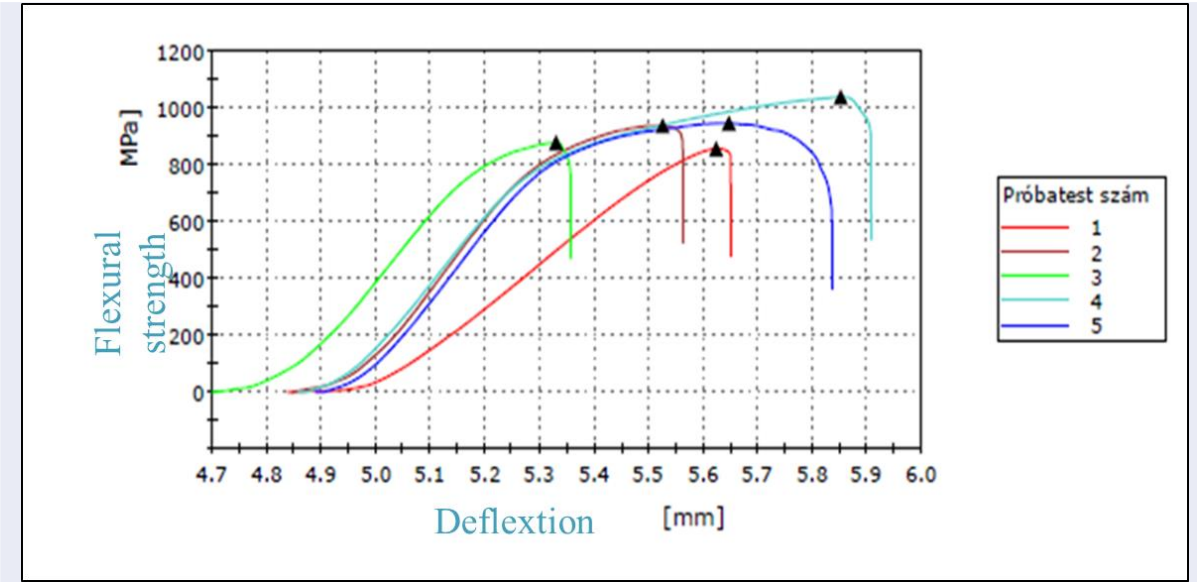
# Three points Bending Test

316L Hognas +**0.33**wt% Si<sub>3</sub>N<sub>4</sub>



Test Number	Sample identifier	Flexural strength	Maximum load
1	0.33wtSi <sub>3</sub> N 1.1	1345	2225,2
2	0.33wtSi <sub>3</sub> N 1.2	1339	2645.1
3	0.33wtSi <sub>3</sub> N 1.3	1301	2601.8
4	0.33wtSi <sub>3</sub> N 1.4	1227	2699.5
5	0.33wtSi <sub>3</sub> N 3.1	1259	2963.5

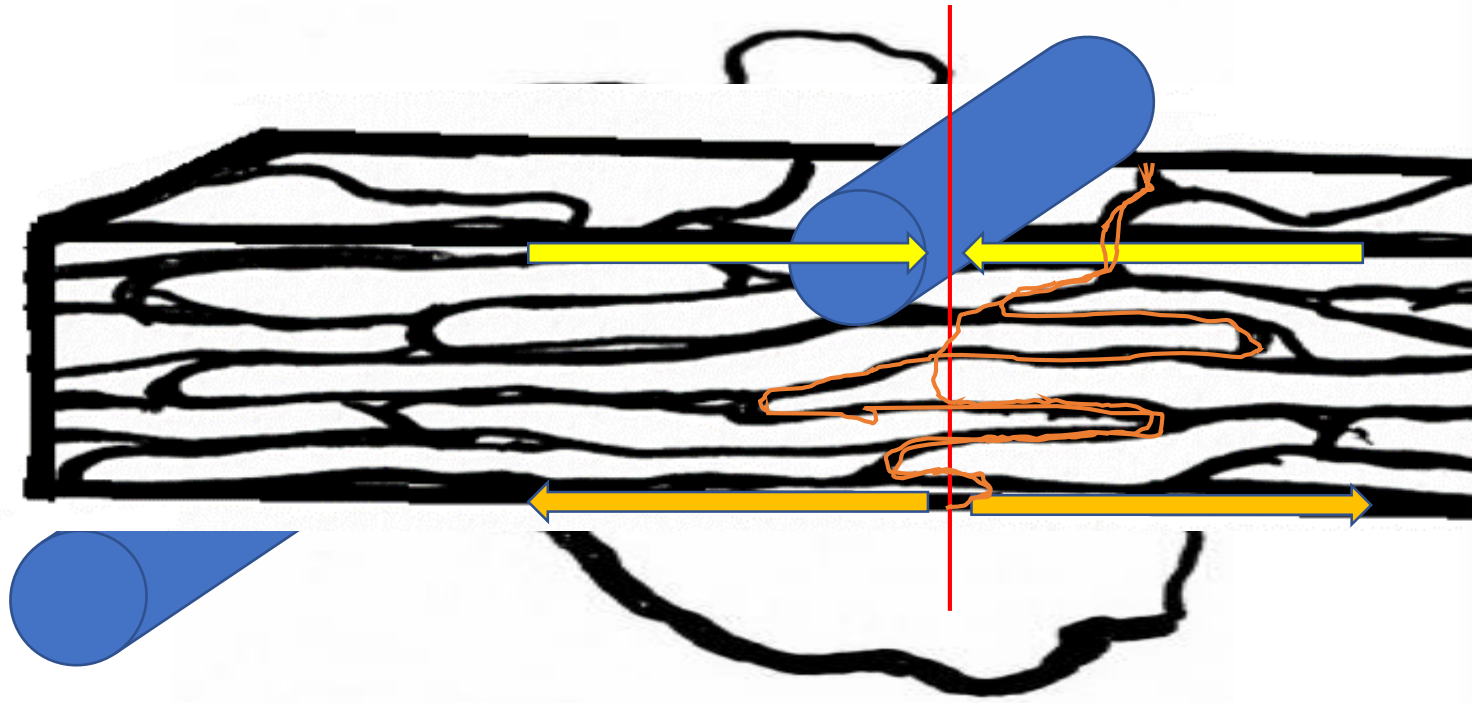
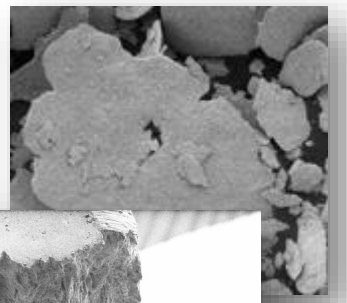
316L Hognas +**1** wt% Si<sub>3</sub>N<sub>4</sub>



Test Number	Sample identifier	Flexural strength	Maximum load
1	1wtSi <sub>3</sub> N4-1	857	4202,7
2	1wtSi <sub>3</sub> N4-4	938	2022,9
3	1wtSi <sub>3</sub> N4-5	878	2087,5
4	1wtSi <sub>3</sub> N4-6	1039	2430,1
5	1wtSi <sub>3</sub> N4-7	947	2168,4

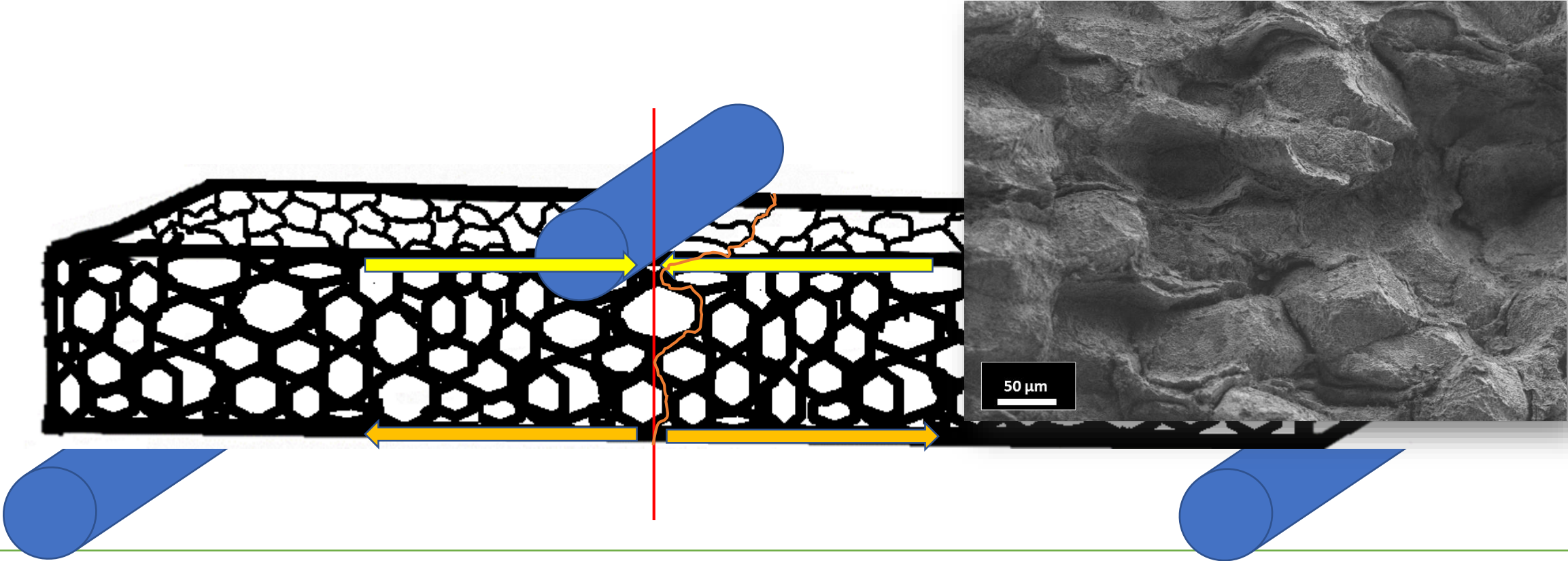
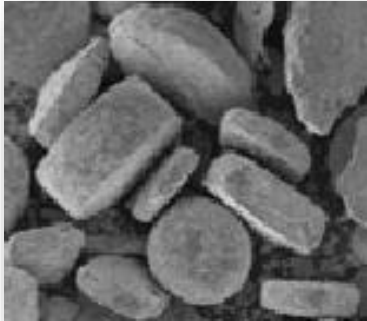


316L Högånäs + 0.33 wt% Si<sub>3</sub>N<sub>4</sub>



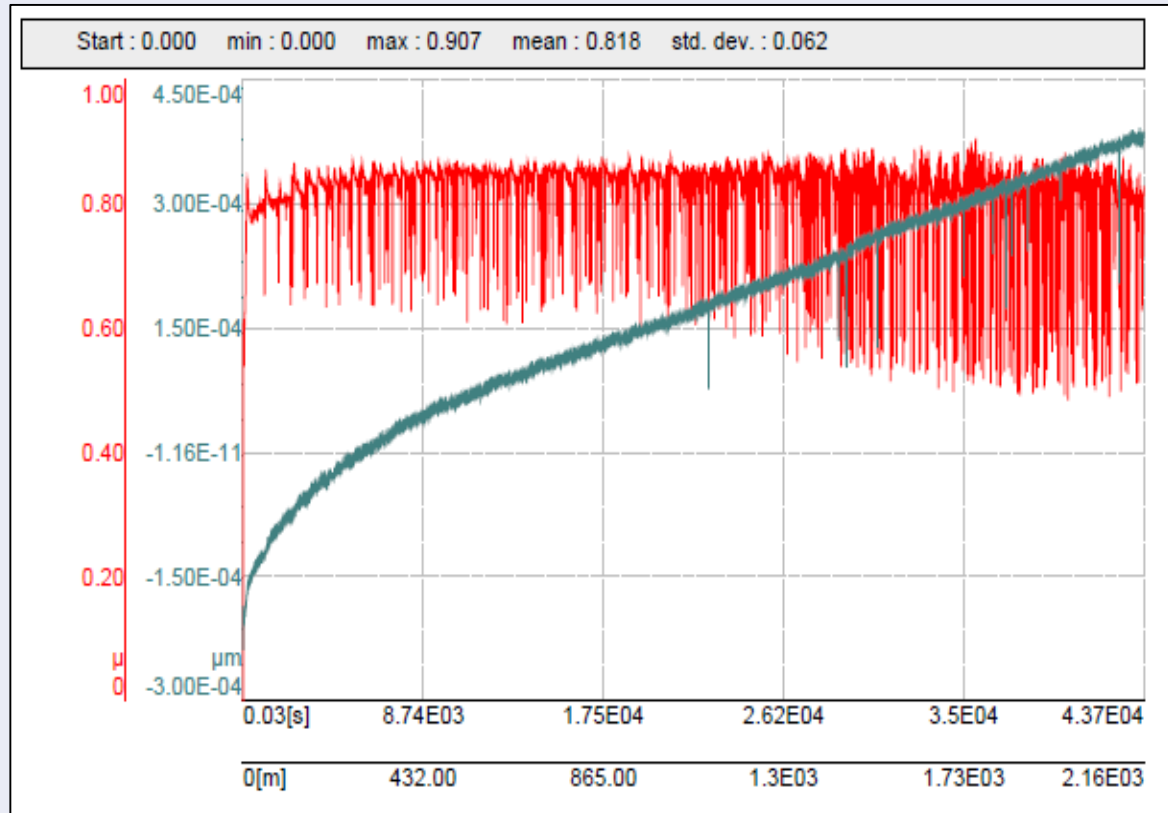


316L Hőganás + 1 wt% Si<sub>3</sub>N<sub>4</sub>



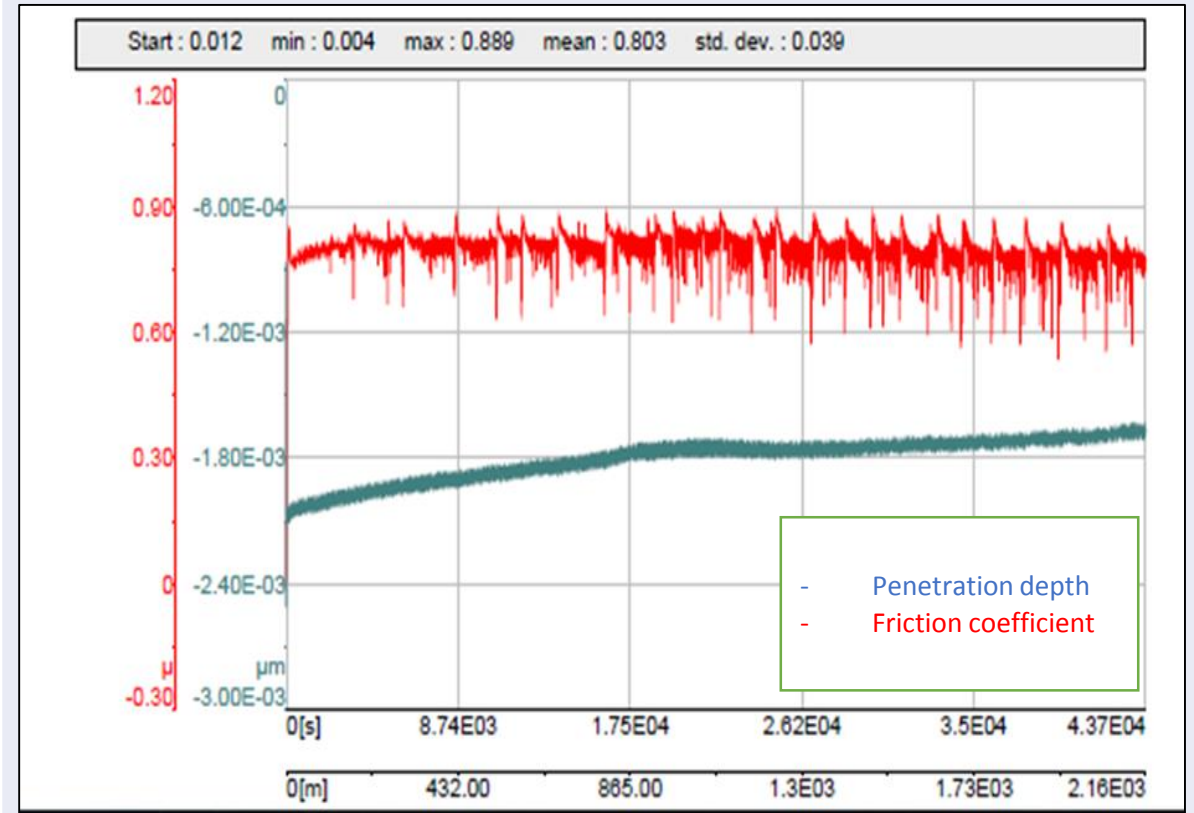
# Tribology

316L Hoganas +0.33wt% Si<sub>3</sub>N<sub>4</sub>

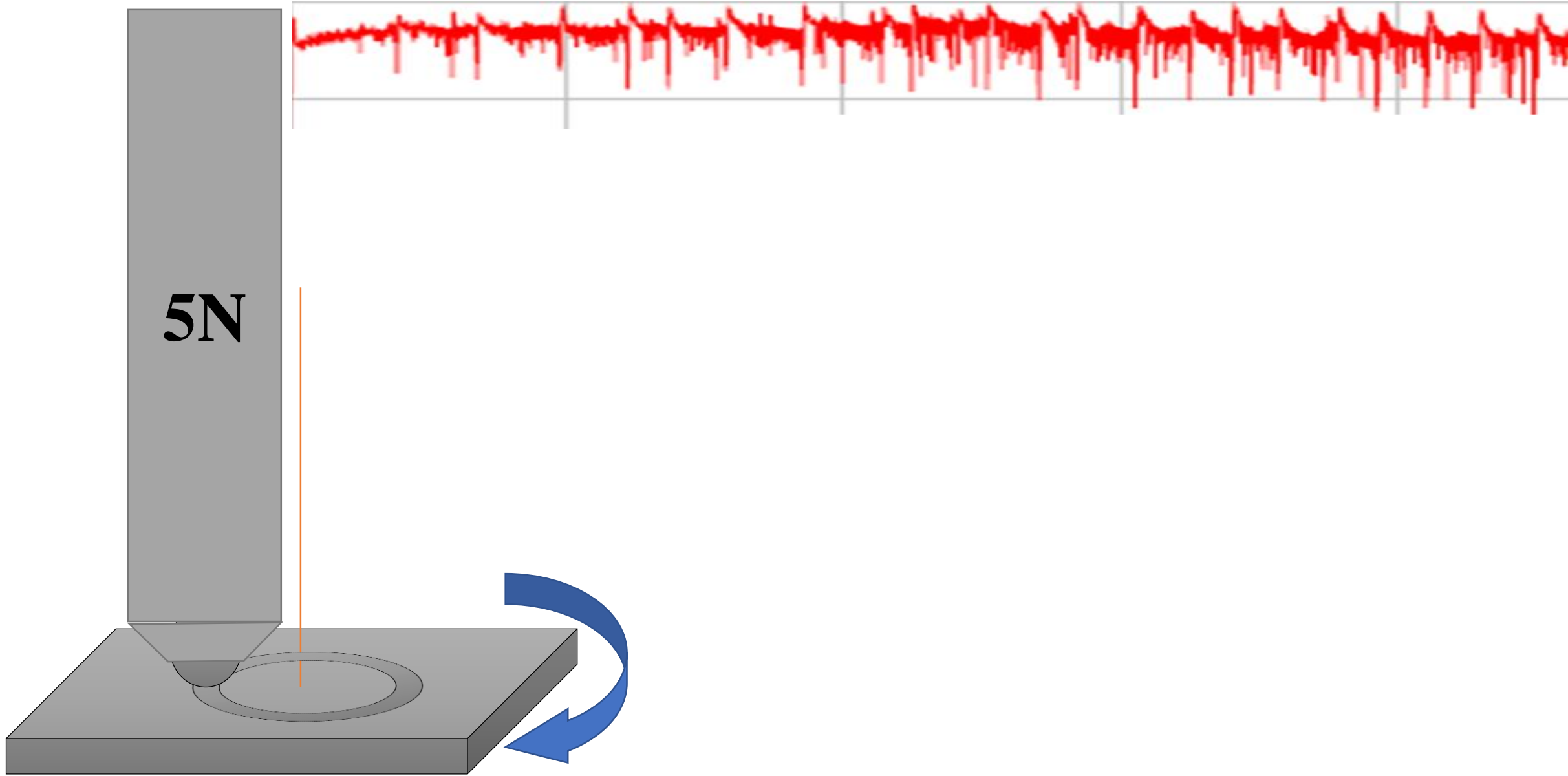


12 hours long tribology test results

316L Hoganas +1 wt% Si<sub>3</sub>N<sub>4</sub>



12 hours long tribology test results



# Conclusions

- Powder technology has been used for ceramic dispersed steel preparation
- 0.33 wt%  $\text{Si}_3\text{N}_4$  addition provided high efficient milling than 1 wt%  $\text{Si}_3\text{N}_4$ 
  - flat 316L grains structure at 0.33 wt%  $\text{Si}_3\text{N}_4$
  - milling effect at 0.33 wt%  $\text{Si}_3\text{N}_4$  -  $\alpha$  Fe presence
- Higher 3 point bending strength at 0.33 wt%  $\text{Si}_3\text{N}_4$  than 1 wt%  $\text{Si}_3\text{N}_4$
- Higher hardness (7.03 GPa) at intergranular parts compared to steel grains (2.53 GPa)
- The friction coefficient was independent of  $\text{Si}_3\text{N}_4$  content

# Results of the Actual semester:

- Preparation of the solid samples for investigation( mechanical surface preparation, water jet cutting, Making modifications on the cutting machine, Making polishing sample holder).
- Structural investigation of the samples ( XRD, EDS, SEM and TEM).
- Learning how to use the tribology test apparatus
- Mechanical investigation of the samples ( three points bending test).
- Participating in the first seminar on “thin films and their applications” at Mohamed khider University, Biskra in Algeria by Oral Presentation (40 minutes) on the 16/04/2017
- Participating in MMT conference in Siofok with Oral Presentation 13/05/2017 (20 minutes)
- Preparing an article to be published in “resolution and discovery” journal
- Preparing a poster for ECERs conference in July 2017, Budapest Hungary

**Taken Subjects:**

**UNFORTUNATELY I DON'T HAVE ☹**

# Results after the second year:

- Participating in SMINS-4 in Manchester by poster & **short oral presentation** ,H.R. Ben Zine, C. Balázs, K. Balázs, A. Horváth, Development of nanostructured ODS steels by powder technology, NEA International Workshop on Structural Materials for Innovative Nuclear Systems, 11-14 July 2016, Manchester, UK, **Poster.**
- Attending the MMT (Hungarian Microscopy Society) Conference in Siófok, 2016. May 19-21
- Participating in the 14th International Symposium on Novel and Nano Materials, 2016. July 3-8,H.R. Ben Zine,  
- F.S. Cinar, O. Yucel, K. Balázs, A. Horváth, C. Balázs, Preparation and Investigation of Boron Nitride Dispersion Strengthened Steels, **presentation.**
- Participating in “ SIXIEME ECOLE SUR LES TECHNIQUES DE CARACTERISATION DES MATERIAUX ”  
by **video conference**
- Participating in **Webinar** conference about « organizing research work and time »
- Submitting a **paper** in “ Courier de Savoir” Journal (1.16 UIF (Universal Impact factor)).
- Participating in the first seminar on “thin films and their applications” at Mohamed khider University, Biskra in Algeria by Oral **Presentation (40 minutes)** on the 16/04/2017



# Results after the second year:

- Participating in MMT conference in Siofok with **Oral Presentation** 13/05/2017 (**20 minutes**)
- Preparing **an article** to be published in “resolution and discovery” journal
- Preparing a **poster** for ECERs conference in July 2017, Budapest Hungary

# Future work Plan:

- Continuing the investigation the  $\text{Si}_3\text{N}_4$  Alloys
- Prepare a paper to be published in high impact factor from the obtained results (with  $\text{Si}_3\text{N}_4$ )
- Start the investigation of the other alloys (316L+ SiC/  $\text{Y}_2\text{O}_3$ /  $\text{Al}_2\text{O}_3$ )
- prepare other papers for publications

A close-up photograph of a hand giving a thumbs up gesture. The hand is the central focus, with the thumb pointing upwards and the other fingers curled. The background is dark and out of focus, showing what appears to be a person in a dark suit. A semi-transparent black horizontal bar is overlaid across the middle of the image, containing the text.

**THANK YOU FOR YOUR ATTENTION**