End of semester IV Presentation

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

Presented by:
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Under the supervision of:
Dr. Balázsi Csaba
Dr. Balázsi Katalin
Content:

- Experiments work Plan
- First Experimental results:
  - 316L Höganäs+ 0.33wt% Si3N4
  - 316L Höganäs+ 1wt% Si3N4
- Results Comparison
- Conclusion
- Results of the actual semester
- Future work plans
Experiments Work Plan:

- **CHARACTERIZATION**
  - SINTERING PROCESS
    - MIXING PROCESS (attrition milling)
      - 316 L stainless steel
      - Si₃N₄
      - 1 wt% 0.33 wt%
    - SPS
  - TRIBOLOGY, HARDNESS, 3&4 POINTS BENDING...
  - SEM, EDS, XRD and TEM.
  - Neutron radiation
  - Heat treatment

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Structure of based materials

316L Höganäs (reference)

- Fe
- 17 wt% Cr
- 12 wt% Ni
- 2.3 wt% Mo
- 0.1 wt% Mn
- 0.9 wt% Si

UBE Si₃N₄ (addition)

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## High efficient attrition milling:

<table>
<thead>
<tr>
<th>Material</th>
<th>Mass</th>
<th>Grinding material</th>
<th>Container</th>
<th>Milling type</th>
<th>Milling Speed</th>
<th>Milling time</th>
<th>Drying conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Höganas 316 L (SS)</td>
<td>300 g</td>
<td>Stainless steel Balls</td>
<td>Big</td>
<td>Wet</td>
<td>Quantity</td>
<td>603 rpm</td>
<td>5 hours</td>
</tr>
<tr>
<td>Si3N4</td>
<td>3 g</td>
<td>Diameter</td>
<td>Quantity</td>
<td>1400 ml</td>
<td>ethanol</td>
<td>300 ml</td>
<td>100 rpm</td>
</tr>
</tbody>
</table>
Sintering Process: Spark Plasma Sintering (SPS):

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Quantity</th>
<th>Sintering temperature</th>
<th>Sintering time</th>
<th>Number of solid Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L+0.33/1wt% Si3N4</td>
<td>600 g</td>
<td>900 °C</td>
<td>5 minutes</td>
<td>1</td>
</tr>
</tbody>
</table>
316L Höganäs + 0.33 wt% Si₃N₄
Structure of milled 316L / 0.33 wt% Si₃N₄

SEM image of milled powder with lamellar grains

XRD spectrum of milled powder

Si₃N₄ is under detection limit

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Structure of sintered 316L / 0.33 wt% Si₃N₄

EDS spectra of the 316L+ 0.33 wt % Si₃N₄ Sintered Sample

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Properties of sintered 316L / 0.33 wt% Si$_3$N$_4$

3 point bending strength measurements

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

HV ~ 3.185 ± 0.41 GPa

Friction coefficient $\mu$ ~ 0.8

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316L Höganäs + 1 wt% Si$_3$N$_4$
Structure of milled 316L / 1 wt% Si$_3$N$_4$

SEM image of milled powder with lamellar grains

XRD spectrum of milled powder

Si holder

Si$_3$N$_4$ is under detection limit

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Structure of sintered 316L / 1 wt% Si₃N₄

SEM image of fracture surface of sintered sample

EDS spectra of the 316L + 1wt % Si₃N₄ Sintered Sample

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Properties of 316L / 1 wt% Si$_3$N$_4$

- **Flexural strength**
  - 3 point bending strength measurements
  - 3p bending strength $\sim 932$ MPa (*5% error)

- **HV ~ 7.03 ± 0.41 GPa**
- **HV ~ 2.53 ± 0.17 GPa**

- **Friction coefficient $\mu \sim 0.8$**
- **SEM image of sample after tribology test**
- **SEM image of indentation**

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Elemental map of sintered 316 L / 1 wt% Si₃N₄
Results Comparison
Morphology after Milling Process

<table>
<thead>
<tr>
<th>316L Hoganas +0.33wt% Si3N4</th>
<th>316L Hoganas +1 wt% Si3N4</th>
</tr>
</thead>
</table>

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XRD

316L Hoganas +0.33 wt% Si3N4

Comparison: as received, Powder Mixture & sintered Sample
- After Milling
- After Sintering
- as received
  - $\gamma$-Fe$_3$Ni: JCPD 03-065-5131
  - $\alpha$-Fe: JCPD 03-066-4899
  - Si holder

316L Hoganas +1 wt% Si3N4

Comparison: as received, Powder Mixture & sintered Sample
- $\gamma$-Fe$_3$Ni: JCPD 03-065-5131
- Si holder
  - after Sintering
  - after milling
  - as received

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Three points Bending Test

316L Hoganas +0.33 wt% Si3N4

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Sample identifier</th>
<th>Flexural strength</th>
<th>Maximum load</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.33wtSi3N 1.1</td>
<td>1345</td>
<td>2225.2</td>
</tr>
<tr>
<td>2</td>
<td>0.33wtSi3N 1.2</td>
<td>1339</td>
<td>2645.1</td>
</tr>
<tr>
<td>3</td>
<td>0.33wtSi3N 1.3</td>
<td>1301</td>
<td>2601.8</td>
</tr>
<tr>
<td>4</td>
<td>0.33wtSi3N 1.4</td>
<td>1227</td>
<td>2699.5</td>
</tr>
<tr>
<td>5</td>
<td>0.33wtSi3N 3.1</td>
<td>1259</td>
<td>2963.5</td>
</tr>
</tbody>
</table>

316L Hoganas +1 wt% Si3N4

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Sample identifier</th>
<th>Flexural strength</th>
<th>Maximum load</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1wtSi3N4-1</td>
<td>857</td>
<td>4202.7</td>
</tr>
<tr>
<td>2</td>
<td>1wtSi3N4-4</td>
<td>938</td>
<td>2022.9</td>
</tr>
<tr>
<td>3</td>
<td>1wtSi3N4-5</td>
<td>878</td>
<td>2087.5</td>
</tr>
<tr>
<td>4</td>
<td>1wtSi3N4-6</td>
<td>1039</td>
<td>2430.1</td>
</tr>
<tr>
<td>5</td>
<td>1wtSi3N4-7</td>
<td>947</td>
<td>2168.4</td>
</tr>
</tbody>
</table>
316L Höganäs + 0.33 wt% Si₃N₄
316L Höganäs + 1 wt% Si$_3$N$_4$
Tribology

316L Hoganas +0.33 wt% Si3N4

12 hours long tribology test results

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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:


- Attending the MMT (Hungarian Microscopy Society) Conference in Siófok, 2016. May 19-21


- Participating in “SIXIEME ECOLE SUR LES TECHNIQUES DE CARACTERISATION DES MATÉRIAUX” 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 Si$_3$N$_4$ Alloys
- Prepare a paper to be published in high impact factor from the obtained results (with Si$_3$N$_4$)
- Start the investigation of the other alloys (316L+ SiC/ Y$_2$O$_3$/ Al$_2$O$_3$)
- prepare other papers for publications
THANK YOU FOR YOUR ATTENTION