EuroHIP Open Meeting

Co-Chairs
Susan Davies (Bodycote HIP AB)  Jim Shipley (Quintus Technologies AB)

Tuesday 16th October
16h45 – 18h15
Overview

- EPMA activities
  Lionel Aboussouan, EPMA

- Trend Survey summary
  Christophe Broeckmann, RWTH Aachen

- Club Projects summary
  Christophe Broeckmann, RWTH Aachen

- Industrial use of PM HIP Technology
  Freddy Busschaert, Total

- HIP Standards
  - A proposed Swedish National Standard and best practice for detection of Ar in PM HIP Material
  Pelle Melin, Swerea Kimab
  - European standards
  Freddy Busschaert, Total
EPMA Secretariat

Dr Lionel Aboussouan  Executive Director

Dr Olivier Coube  Technical Director

Mr Andrew Almond  Marketing & Exhibition Manager

Miss Kate Blackbourne  Congress Manager

Mrs Jackie Peters  Accounts

Mrs Delphine Nicolas  Event and Project Co-ordinator

Mrs Sabine Hazoumé  Event and Project Co-ordinator

Mrs Rhianna Jones  Membership, Database & IT Co-ordinator

Mr Andy Cormack  Marketing Assistant

Miss Emma Powell  Event Assistant

Ms Karen Fisher  Graphic Designer

www.epma.com/meet-the-team
EPMA Website for Case Studies

- www.epma.com/spotlight-on-pm
EPMA Social Media - LinkedIn

- Be connected with EPMA LinkedIn group
- Share post
EuroHIP News 2018

• EuroHIP meeting together with EMPA General Assembly in March
• Trend Survey → need to involve more members
• Key statistic figures released
  • Powder and part production → need to involve more members
• Statistic brochures released
  • Economic
  • Automotive
  • Aerospace
  • Medical
• HIP Seminar: Design for HIP
  • Västerås – Sweden with a site visit to Quintus Technologies facilities
  • 55 participants (Europe, Asia)
EuroHIP News 2019

• EuroHIP Seminar – February 2019 (Next slide)
  • Seminar in Sint-Niklaas, Belgium. Site Visit Hosted by EPSI

• EuroHIP Meeting with General Assembly, 21-22 March in Brussels

• PM Life (Next slide)

• EuroHIP meeting with next EuroPM in Maastricht
  • Special Euro HIP session linked to EPMA 30 years anniversary: the past and future for HIP
2019 HIP Seminar

- February 11-12 – Sint-Niklaas, Belgium
- Conventional HIP and Rapid Cooling HIP: Developments in Material Property and Microstructural Relationships
- Call for Speakers is open
  - seminars.epma.com/hip-2019
- Plant tour to EPSI
PM Life is a new lifelong training programme to help develop the Powder Metallurgy Future.

The course will offer participants chance to either attend the full programme to learn about the whole industry or pick topics to help further their knowledge in certain areas.

The course offers five weeks training and an optional internship covering five strands.

Express your interest by contacting us on RJ@epma.com or completing a short survey at www.surveymonkey.co.uk/r/PMLifeSurvey.

Scan the QR code to enter the survey via your mobile!

www.pmlifetraining.com
HIP Surveys

• Only 1 online survey with different parts (you can skip questions):
  • Production data (ton / €)
  • Trend Survey

• Data are collected December – February

• Express interest in October – November
  • Ensure only 1 person per company
  • Non Disclosure Agreement if needed
  • You can contact Lionel Aboussouan: la@epma.com
Club Project Summary

Presented by Christophe Broeckmann
Institute for Materials Application in Mechanical Engineering (IWM)
RWTH Aachen, Germany
EPMA Club Projects

Industry-defined Projects open to EPMA Members and Co-ordinated by the EPMA

- Budget: Between €10k and €100k
- Time frame: Between 6 and 18 months
- 19 Projects so far: www.epma.com/projects
- €650k collected in total

Club Projects Webpage: www.epma.com/projects

Club Project meeting participants

Funding from Industry Partners

Co-ordination by the EPMA

Club Projects

Project Ideas from Universities, R&D Centres and Contractors

Synergies
AM&HIP 18
Combination of Additive Manufacturing and HIP

Christoph Broeckmann, Anke Kaletsch, Johannes Kunz

Institute of Applied Powder Metallurgy and Ceramics at RWTH Aachen e.V. (IAPK)

Kick-Off Meeting, Düsseldorf, 16.02.2018
Content

1. Motivation
2. Project objectives
3. Work packages
4. Time plan
5. Cost
6. Discussion
Motivation

- L-PBF microstructure and porosity e.g.
  - CoCr alloy F75 as L-PBF
    - relative density: 99.7%
  - Hot working steel H13 as L-PBF
    - relative density: >99.6%
    - Micro cracks

- L-PBF + HIP microstructure and porosity e.g.
  - CoCr alloy F75 as L-PBF
    - relative density: 100%
  - Hot working steel H13 as L-PBF
    - relative density: 100%
    - No micro cracks
Motivation

- Endurance limit of alloy F75 in different production routes
  - as cast: $\sigma_{A50} = 296$ MPa
  - as L-PBF: $\sigma_{A50} = 123$ MPa
  - L-PBF+HIP: $\sigma_{A50} = 470$ MPa

![Endurance limit graph](image-url)
Motivation

• The costs for L-PBF scale with the scanning speed of the laser beam.

• The speed can be increased when no full density is required.

• Post densification by HIP reduces porosity

=> Combination of L-PBF + HIP gives a potential for an optimum in terms of production costs
Objectives

1. Identification of a set of HIP parameters to obtain full density and optimal mechanical properties in components produced by L-PBF

2. Determination of the increase in fatigue strength that can be obtained by adding a HIP cycle to the L-PBF-process

3. Optimization of the entire process chain: L-PBF + HIP in order to get a solution with minimum process costs and maximum performance regarding mechanical properties
1. Identification of a set of HIP parameters to obtain full density and optimal mechanical properties in components produced by L-PBF

2. Determination of the increase in fatigue strength that can be obtained by adding a HIP cycle to the L-PBF-process

3. Optimization of the entire process chain: L-PBF + HIP in order to get a solution with minimum process costs and maximum performance regarding mechanical properties
Work packages

WP 0  Powder supply  IWM

WP 1  Production of specimens by SLM  IWM

WP 2  Post densification by HIP  IWM

WP 3  Heat treatment  IWM

WP 4  Characterization of density and microstructure  IWM

WP 5  Fatigue tests  IWM
WP0: Powder Supply

• two grades to be specified by the industrial consortium, for example
  • Steel base
    • Tool steel (H13, M2)
  • Super Alloy
    • IN718

• Powder to be supplied by industry
WP1: production of specimens by L-PBF

• iterative identification of optimal L-PBF-parameters by cube shaped specimens
  • available L-PBF machine: Realizer SLM100

• production of cube shaped specimens with different density
  • due to variation of L-PBF parameters

• production of specimens for fatigue tests (rotating-bending tests)
  • specimens are machined to the final sample geometry, assuring equal surface quality for all samples

Scan velocity
WP2: post densification by HIP

- Optimal set of HIP-parameter to obtain full density

- Variation of HIP parameter
  - Pressure,
  - Temperature
  - Dwell time

- 12 HIP cycles
  - 6 per material
WP3: Heat treatment

• depending on the particular material:
  • no heat treatment
  • harden and temper (tool steel)
  • solutionize (austenitic steel, duplex steel)
  • solutionize + aging (super alloy, maraging steel)
WP4: characterisation of density and microstructure

- density measurement
  - He-pycnometry and Archimedes principle

- Light optical micrographs in order to determine
  - porosity
  - grain size

- SEM in order to identify crack origins in the fracture surface

- Comparison with and without HIP post treatment
WP5: Fatigue tests

- rotating bending tests
  - Stress ratio $R = -1$

- for each grade:
  - 2 S-N-diagrams (Wöhler-curve)
    - statistics: 30 specimen each

- Comparisson of endurance limits of two sta
  - as L-PBF
  - as L-PBF + HIP
## Work package time planning

- **Duration of the project:** 10 months

<table>
<thead>
<tr>
<th>Work Package</th>
<th>Duration</th>
<th>Month</th>
</tr>
</thead>
<tbody>
<tr>
<td>WP0 Powder supply</td>
<td>1-2</td>
<td>1-2</td>
</tr>
<tr>
<td>WP1 Production of specimens by L-PBF</td>
<td>3-5</td>
<td>3-5</td>
</tr>
<tr>
<td>WP2 Post densification by HIP</td>
<td>6-8</td>
<td>6-8</td>
</tr>
<tr>
<td>WP3 Heat treatment</td>
<td>9-10</td>
<td>9-10</td>
</tr>
<tr>
<td>WP4 Characterization of density and microstructure</td>
<td>1-10</td>
<td>1-10</td>
</tr>
<tr>
<td>WP5 Fatigue tests</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Reporting</td>
<td>KM</td>
<td>IM</td>
</tr>
</tbody>
</table>

**KM:** Kick-off meeting,  
**IM:** Interim Meeting,  
**FM:** Final Meeting
## Cost figure

<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>IAPK personnel: 0.2 full engineer for 10 months:</td>
<td>€10,400</td>
</tr>
<tr>
<td>0.3 technician for 8 months:</td>
<td>€9,750</td>
</tr>
<tr>
<td>1.0 Student for 10 months:</td>
<td>€5,500</td>
</tr>
<tr>
<td>Overheads on personnel:</td>
<td>€15,390</td>
</tr>
<tr>
<td>Consumables (metallography, L-PBF, fatigue testing)</td>
<td>€6,000</td>
</tr>
<tr>
<td>HIP cycles (personnel included above)</td>
<td>€6,000</td>
</tr>
<tr>
<td>Travel costs (3 meetings à 2 persons)</td>
<td>€2,400</td>
</tr>
<tr>
<td><strong>Total funding:</strong></td>
<td>€55,440</td>
</tr>
<tr>
<td>EPMA administrative costs (15%)</td>
<td>€8,316</td>
</tr>
<tr>
<td><strong>Total costs of project</strong></td>
<td>€63,716</td>
</tr>
</tbody>
</table>

All costs exclusive VAT if applicable
Thank you for your attention!

Christoph Broeckmann, Anke Kaletsch, Johannes Kunz

IAPK – Institut für Anwendungstechnik Pulvermetallurgie und Keramik
an der RWTH Aachen e.V.
Augustinerbach 4
52062 Aachen

www.iapk.rwth-aachen.de
Industrial use of PM HIP Technology

Freddy Busschaert, Total
Industrial use of PM HIP Technology

- Freddy BUSSCHAERT,
- DSO/ DeepWater & Subsea
- TOTAL E&P ,
- PAU, France
Industrial Use of PM Hip Technology

• Challenges of deep offshore developments
• Advantages in the use of PM HIP components
• Duplex Stainless Steels
• Complex shaped components for Deep offshore
• HP /HT topside PM Hipped components
Challenges of deep offshore developments

• The need of High performance materials
  • High strength mechanical properties
  • External and internal corrosion resistance

• High Pressure / High Temperature environments

• Hydrogen Embrittlement - Subsea Failures

• Heavy wall and complex shaped components
Deep offshore development overview
Advantages in the use of PM HIP components

• HIP is a field-proven manufacturing process
• Fine microstructure / Isotropic mechanical properties
• Good resistance to HISC (Hydrogen Induced Stress Cracking)
• Complex shaped components
• Near Net Shape components
• Good weldability
• Less welds and associated NDE to be performed
• An efficient supply chain which allows Hip components to be delivered within a reasonable timeline (14 weeks average)
Duplex and Super duplex Stainless Steels

- Intensive use of:
  - Duplex Stainless Steel (ASTM A988 UNS S31803)
  - Superduplex Stainless Steel (ASTM A988 UNS S32505)

- Minimum Yield strength (450 MPa for 22Cr and 550MPa for 25Cr)
### Duplex and Superduplex Stainless Steels

Duplex and Superduplex Stainless Steels present a mixed microstructure of austenite and ferrite phases (usually a ratio of 40/60)

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>W</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNS 31803</td>
<td>0.03</td>
<td>22</td>
<td>4.5</td>
<td>3</td>
<td>-</td>
<td>-</td>
<td>0.2</td>
</tr>
<tr>
<td>UNS 32505</td>
<td>0.03</td>
<td>25</td>
<td>7</td>
<td>3.5</td>
<td>0.5</td>
<td>0.5</td>
<td>0.2</td>
</tr>
</tbody>
</table>
Fine grained microstructure

Austenite spacing value estimated to 12 microns
Hipped hubs in Super duplex Stainless Steel on DALIA project (2006)

- Manufacturing of those hubs by the HIP process instead of forgings
Typical complex shaped components
Typical complex shaped components
Pump casing and flange
Hubs
Manifold Headers and Wye piece
Hipped Headers 14 inches
Manifolds
Pazflor Subsea separation
HP /HT topside PM Hipped components
18" Manifolds
10” Manifold with circumferential welds complete
progress on 10” circumferential weld
Thanks for your attention
HIP Standards
A Proposed Swedish National Standard And Best Practice For Detection Of Ar In PM HIP Material

Pelle Mellin, Otto Björnberg, Ingrid Bengtsson, Daniel Gonzalez, Martin Östlund, Henrik Blom, Jenny Höglund, Saeed Klamehr

pelle.mellin@swerim.se
Swerim – Swedish Research Institute for Mining, Metallurgy and Materials

A new industrial research institute within mining engineering, process metallurgy, materials and applications, formed on 1 October 2018.

- Swerim AB is formerly known as Swerea KIMAB (and Swerea MEFOS)
- Around 190 employees
- Business in Luleå and Stockholm
- Approx. 250 million kronor in turnover
Contents

• Argon porosity
• Argon porosity in PM HIP
• Prior work on detection of Ar
• The proposed standard
• Best practice for Ar detection
• Conclusions
• Acknowledgments
Argon porosity

3D visualisation of the porosity (red) imaged by CT scans of the same cylindrical sample (build direction vertical) (a) as-built; (b) following HIPing; (c) 10 min at 1035 °C; (d) 10 h at 1035 °C; and (e) 10 min at 1200 °C. [1]

Argon porosity in PM HIP

Start of HIP:
- Welded can
- Metal powder
- Consolidation front
- High isostatic pressure

End of HIP:
- Small round pores
- High isostatic pressure

Heat treatment:
- Expanded angular pores
Argon porosity in PM HIP Alloy with good hot-strength

Heat treatment at 1060 °C for 3 h, Ar content 48 ng/g
Argon porosity in PM HIP Alloy with good hot-strength

Heat treatment at 1200 °C for 3 h, Ar content 48 ng/g
Argon porosity in PM HIP Alloy with less hot-strength

Heat treatment at 1060 °C for 3 h, Ar content 1637 ng/g
Argon porosity in PM HIP Alloy with less hot-strength

Heat treatment at 1200 °C for 3 h, Ar content 1637 ng/g
Circularity of pores as function of pore diameter

- Circularities range from 0 to 1.
- Feret's diameter is measured in micrometers (μm).
- The graph shows data points for all pores and a fitted Power curve.
Impact toughness

Charpy-V specimen 10 x 10 x 55 mm

Before HIP
After HIP (30% shrink)

133.19 mm
Top view

88.79 mm

Front view

60.93 mm
30.47 mm

60,93 mm
30,47 mm

88,79 mm

150 mm
Impact toughness

Charpy-V impact toughness at room temperature, as function of Ar concentration. In our study, six capsules filled with 316L powder (0-500 μm) was partially evacuated.

Prior work on detection of Ar
Prior work on detection

• Hofer et al [1, 2] presented methodology for detection of Ar using MS (G8 Galileo).

• Broeckmann et al [3] described some details on detection of Ar using GC (Eltra-WERF).


The proposed standard
or rather... the already published standard
The published standard

- SS 118000:2018
- Title: Powder metallurgy - Hot isostatic pressing - Argon detection using gas chromatography and mass spectrometry techniques
- A Swedish national standard
- Only in English
- Available for purchase on https://sis.se/produkter/metallurgi-dfbdd22b/pulvermetallurgi/ss-1180002018/
- Includes only PM HIP material
- Excludes AM material that may also suffer from Ar porosity
- Considers the distinctiveness of PM HIP
- Most importantly the standard encourages sampling that considers segregation of Ar.
The published standard

Test piece
Test sample (free from can material)
Rinsing in ethanol
Drying
Loaded via purged sample port
Degassing
He
Graphite crucible
Ready for analysis
Inert gas fusion
He
GC or MS
Ar
The published standard

The standard mandates that the test piece shall be removed from either a sacrificial part or an integrated part of the HIP canister.
Sampling of a test piece

Sampling from corners:

Sampling from filling pipe:

Main body of PM HIP can

Test piece

Test sample

Machined volume

Sampled material

Pipe powder

Test sample

Filling pipe

Main body of PM HIP can

Test piece

Test sample
The published standard

The standard mandates that a test sample shall be free from can material and the mass shall be between 0.5 and 0.9 g.

Also the sample shall be one single piece with a non-slender geometry.
The published standard

Reduces soot and grime in the instruments and reduces the time for drying

Test piece
Test sample (free from can material)
Rinsing in ethanol
Drying
Ready for analysis
Inert gas fusion

Graphite crucible
Degassing

He

Loaded via purged sample port

He

Ar

GC or MS
The published standard

No specific instructions other than that the sample should be completely dry afterwards.
Weigh, and either load the sample via a purged sample port or manually place the sample in a crucible.

Test piece
Test sample (free from can material)
Rinsing in ethanol
Drying
Degassing
Loaded via purged sample port
Ready for analysis
Inert gas fusion
GC or MS
The published standard

Heating of the test samples rapidly to approximately 2200 °C, under a flow of He (purity ≥99.9999 %)
Test procedure, standard

• Calibration
• Test of blank sample
• Test of reference sample
• Perform the test
• As a minimum, record in the test reports the following information:
  • All details necessary for the identification of the test sample’s parent PM HIP component.
  • Expression of the result as above or below the limit agreed upon between the purchaser and provider of the PM HIP service.
  • Date of test, authorized signature, test equipment and laboratory identification.
Reference samples

Using regulated air pressure [1]

Using microspheres [2]


Best practice for Ar detection

The basis for the best practice is a survey carried out by Swerim which included visits to Sandvik Powder Solutions AB in Surahammar, Bodycote HIP AB in Surahammar and Erasteel Kloster AB in Söderfors. Note that only gas chromatography is covered by the best practice.
Best practice

These two sampling methods work equally well according to results not presented here. Other methods exist, that are covered by the standard, but not by the best practice study.
Best practice

best practice: remove skin material as well, 0.8 g mass

best practice: weigh accurately and use an instrument with an integrated purged sample port.

Graphite crucible

Degassing

He

Loaded via purged sample port

He

GC or MS

Inert gas fusion

best practice: calibration test of 2 blank samples
test of 2 reference samples test
Test procedure, best practice

- Calibration
- Test of 2 blank samples
- Test of 2 reference samples, one high Ar level, one low
- Perform the test
- As a minimum, record in the test reports the following information:
  - All details necessary for the identification of the test sample’s parent PM HIP component.
  - Expression of the result as above or below the limit agreed upon between the purchaser and provider of the PM HIP service.
  - Date of test, authorized signature, test equipment and laboratory identification.
Conclusions

- This study found a similar relationship between impact toughness and Ar content (for 316L) as Broeckmann et al [1].

- The users of GC and MS have accumulated experience over the years and consider them robust. Consensus has also been reached on how to sample reliably. Since consensus has emerged, a standard is now possible.

- We firstly propose a national standard that is now published. It states minimum requirements. The proposed standard considers the distinctiveness of PM HIP. For example that Ar tends to segregate in the PM HIP can.

- We present also a best practice study, which represents the most accomplished procedure found during visits to a number of Swedish PM HIP producers.

Acknowledgments

• This research was funded through the Membership Research Consortia (MRC) at Swerim.
• Supporting companies are:
  • AB Sandvik Materials Technology
  • Bodycote HIP AB
  • Erasteel Kloster AB
  • Carpenter Powder Products AB.
• The authors would like to thank all reviewers of the standard and in particular Staffan Thurfjell (at TAKON) and Peter Papelewski (at Bruker) for answering questions regarding their GC and MS-based techniques respectively.
• The foundation Axel Hultgren is acknowledged for a travel scholarship used to attend this conference.
Pelle Mellin Ph. D
+46 (0)8 440 48 83 | +46 (0)72 584 59 12
pelle.mellin@swerim.se | www.swerim.se | map
ISO/TC 119 Powder metallurgy

• Secretariat Swedish Standards Institute (SIS)
• Secretary Otto Björnberg, SIS (Sweden)
• Chairperson Mats Larsson, Höganäs AB (Sweden)

Scope of ISO/TC 119:
Standardization of powder metallurgical materials concerning terms and definitions, sampling, testing methods and materials specifications.

Structure of ISO/TC 119:
WG 2  Maintenance of ISO 5755 and ISO 22068 (former SC 5 Specifications for powder metallurgical materials (excluding hardmetals)), SIS
WG 3  Revision of ISO 3252, SIS
SC 2  Sampling and testing methods for powders (including powders for hardmetals), SIS
SC 3  Sampling and testing methods for sintered metal materials (excluding hardmetals), DIN
SC 4  Sampling and testing methods for hardmetals, DIN
(SC = Sub committee, WG = Working group)

Participating members (P-members):
Austria (ASI)  China (SAC)
Germany (DIN)  India (BIS)
Japan (JISC)  Korea, Republic of (KATS)
Russian Federation (GOST R)  Spain (UNE)
Sweden (SIS)  United Kingdom (BSI)
United States (ANSI)
ISO/TC 119 Powder metallurgy

(Resurrection of) SC 5 Specifications for powder metallurgical materials (excluding hardmetals)

In 2015 ANSI/MPIF has asked to be relinquished from as secretariat of ISO/TC119 SC5 and no other national standardization body was at that time willing to take over the secretariat of SC5.

Since then a working group under the secretariat of TC119 have been handling the PM materials standards. For the time being the work to update ISO 5755 has been initiated, the present version is from 2012. There are other areas were PM material standards are needed, a working group has been formed to work on a standard for porous filter materials. Other PM material standards considered are for example for powder forged materials and soft magnetic materials.

To develop new PM material standards and maintain and develop the existing standard is however a too large task to be administrated under at Work group and a Sub committee for PM material standards is needed.
ISO/TC 119 Powder metallurgy

(Resurrection of) SC 5 Specifications for powder metallurgical materials (excluding hardmetals)

The solution that have been proposed is that SC5 is reactivated with the secretariat at UNE (Spanish NSB) and that the chairperson is Jesús Peñafiel, AMES (Spain).

The secretariat of SC5 is suggested to be financed by EPMA with an annual fee of €11 000.
European Standards

Freddy Busschaert,
• TOTAL E&P,
• PAU, France
European Standards

• To day, HIP industry has used intensively standard ASTM 988 as this is the unique standard.

• ASTM A 988 2017 Edition is the latest edition

• Does it satisfy the industry?

• In case of new UNS grade to be added, does it take a while to be included in the standard?
European Standards

• New Powders to be proposed in the PM industry: any benefit to have an ISO standard which could be easier to update with a new Powder Grade?

• Do you need to have an European Standard as an alternative to the existing ASTM 988 standard?
Thanks for your attention
Before leaving today

• If you were not already in touch with EPMA staff about the EuroHIP group, thanks to leave a business card before you leave the room.