Control of Defects and Microstructure in ODS Alloys

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Control of Defects and Microstructure in ODS Alloys

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- Recrystallisation of PM2000
  - torsionally deformed tube
  - extruded bar
  - sheet samples subjected to bending
  - sheet samples deformed torsionally
- Torsional forming trials on ASTM 446 ferritic steel tube
- Evolution of Oxide Dispersions during Secondary Recrystallisation
- Replacement ODM751
- Selective Laser Melting (SLM) of PM2000 alloy powder

Recrystallisation of torsionally deformed PM2000 tube

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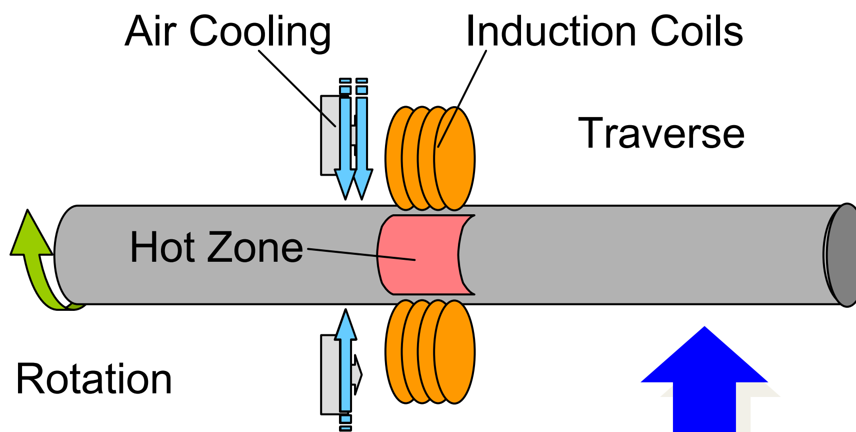
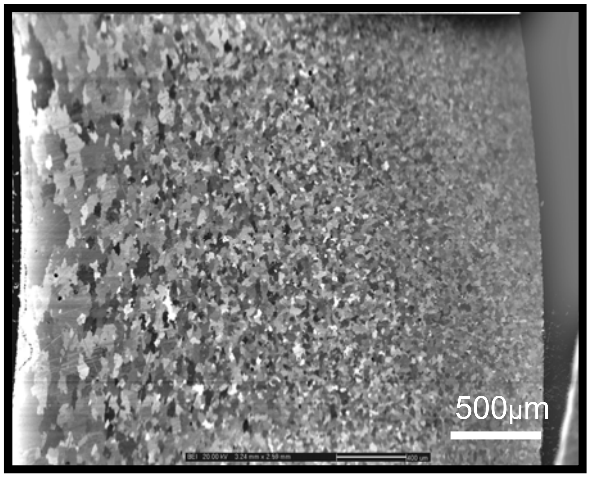


Torsion trial parameters
- 1.25 rpm torsion
- traverse rate, $0.01 ms^{-1}$
- lead length 200mm
- 720^0C
- 20^0 twist

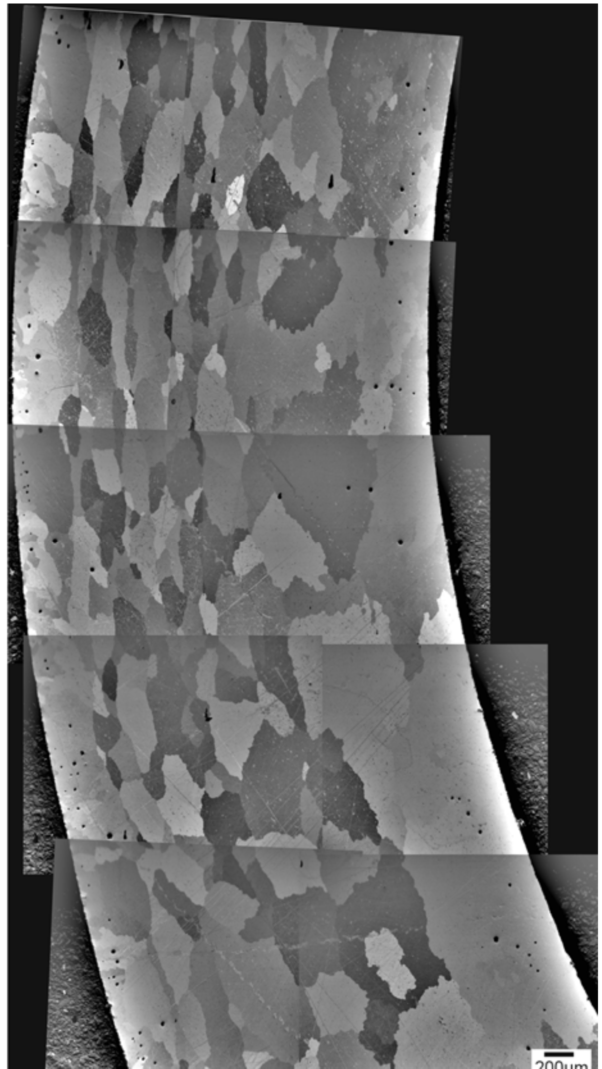
PM2000
- fine grained
- 2 metre length
- 25.8 mm O.D.
- 2.9 mm wall

End-section of 2 meter length of PM2000 tube, twisted at Forecréu at 750^0C.

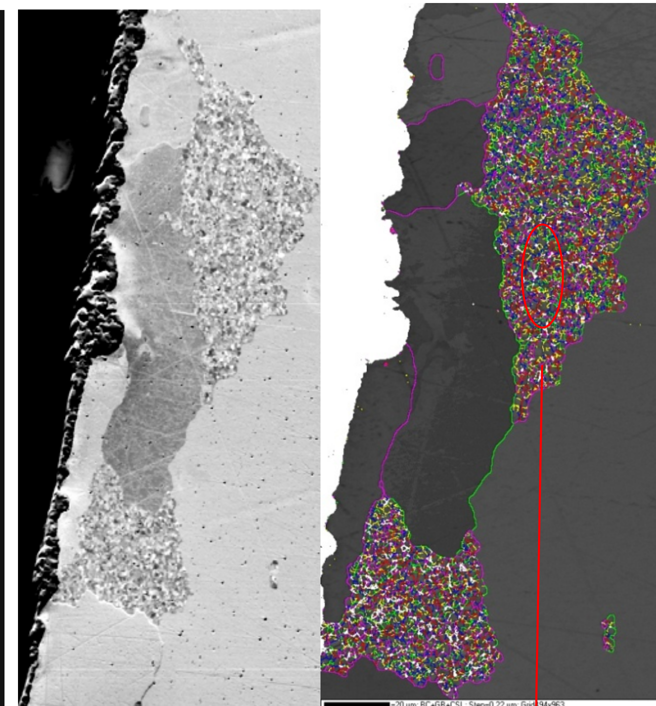
Recrystallisation of torsionally deformed PM2000 tube



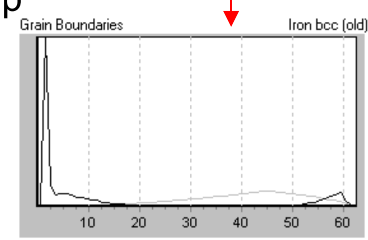
PM2000 tube ‘end stock’
+ SR 1380^{0}C/1h.
Transverse section, SEM



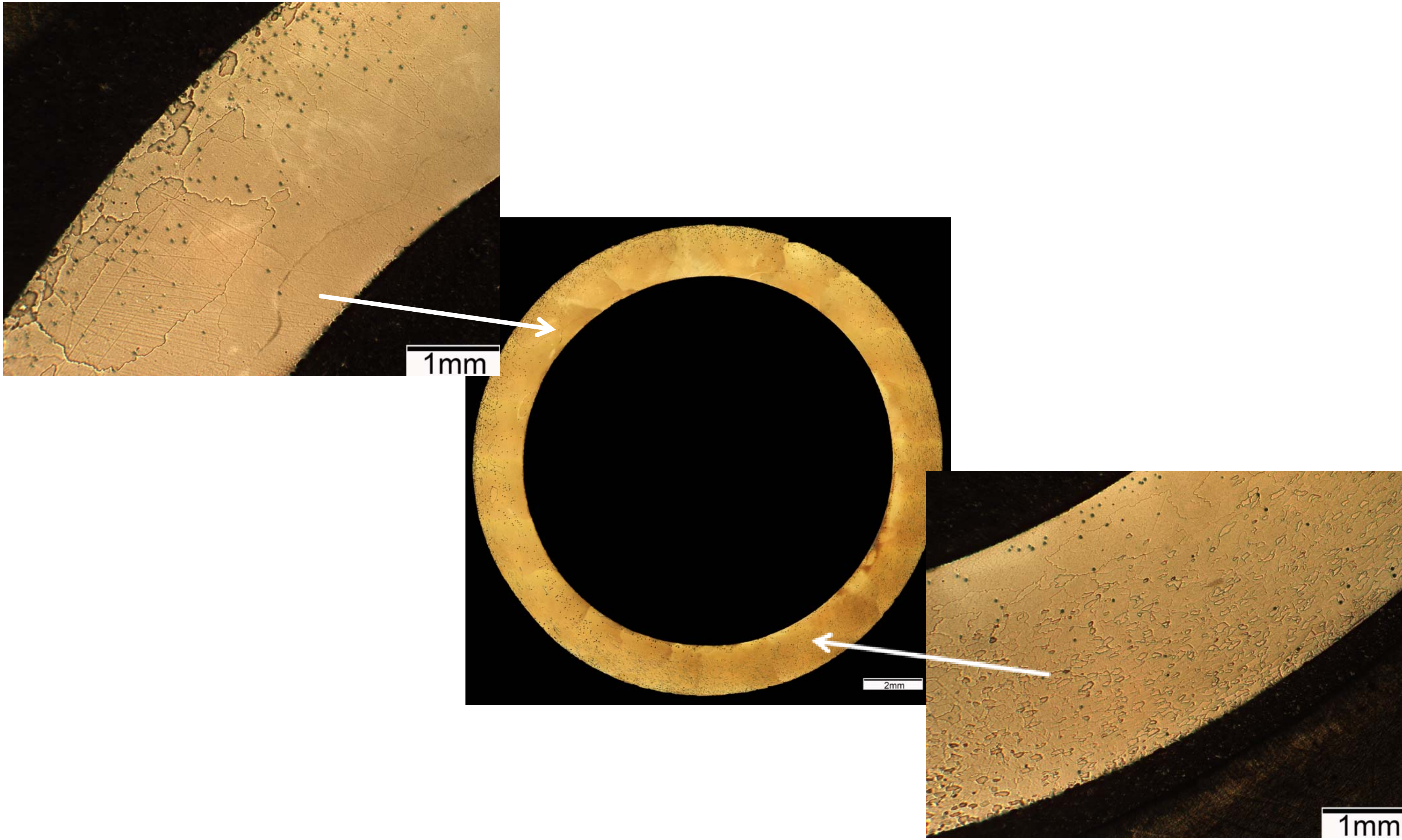
PM2000 tube torsionally deformed ~20%
+ SR 1380^{0}C/1h. Transverse section, SEM.



PM2000 tube torsionally deformed ~20
+ SR 1380^{0}C/1h.
Inner wall. Transverse section, SEM.
a) channelling contrast SEM
b) Boundary map

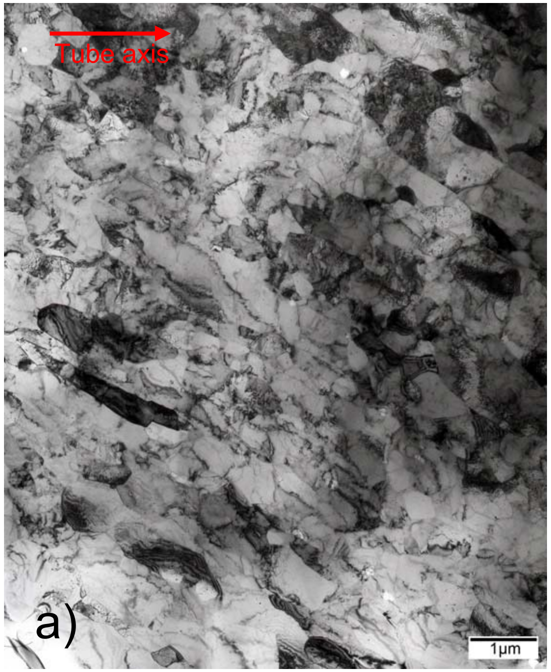


Recrystallisation of torsionally deformed PM2000 tube

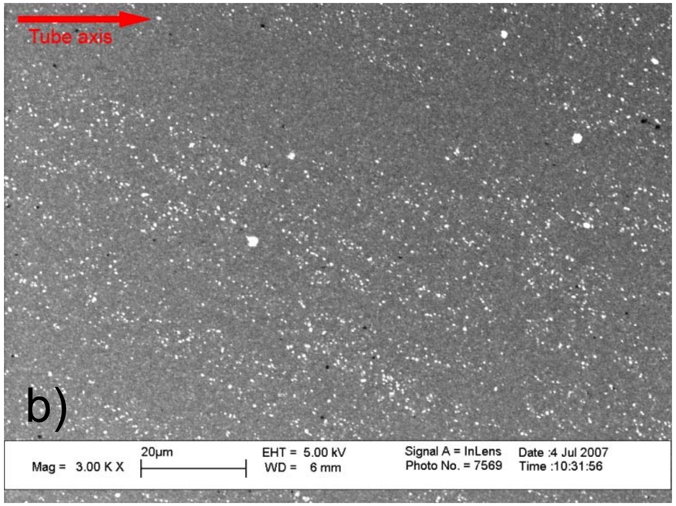


Transverse section through PM2000 tube torsionally deformed + SR 1380^{0}C/1h

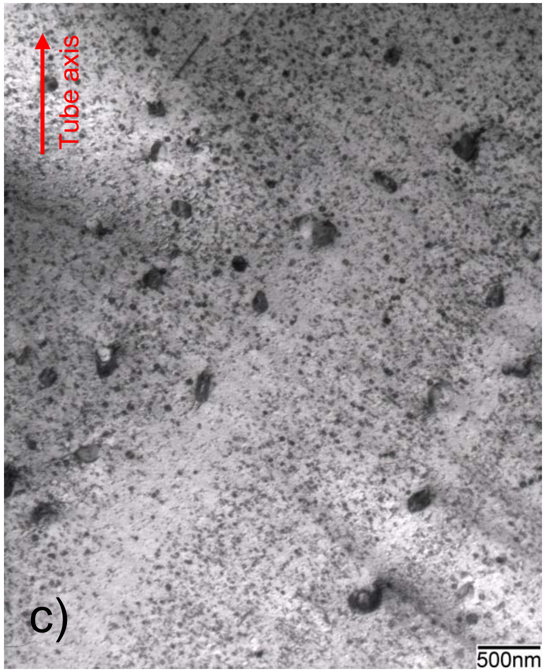
Recrystallisation of torsionally deformed PM2000 tube



a) torsionally deformed ~20%
Plan section. TEM.

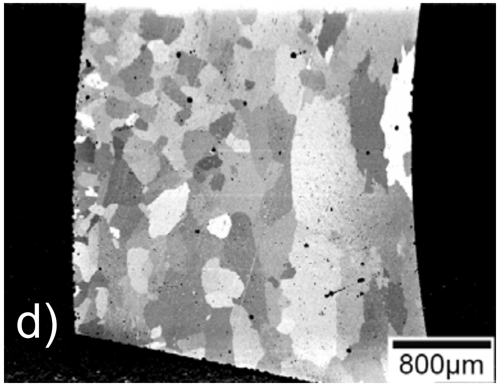


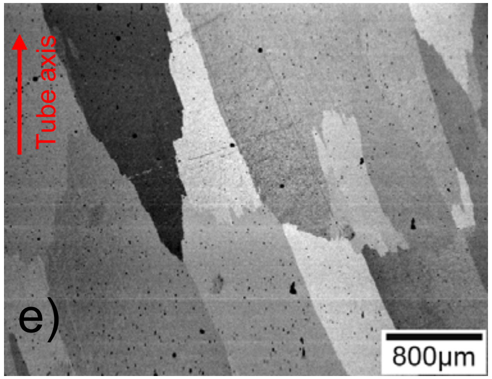
b) Deformed + SR 1380^{0}C/45 min.
Plan section. LVSEM (5kV).



c) Deformed + SR 1380^0/1h.
Plan section. TEM.

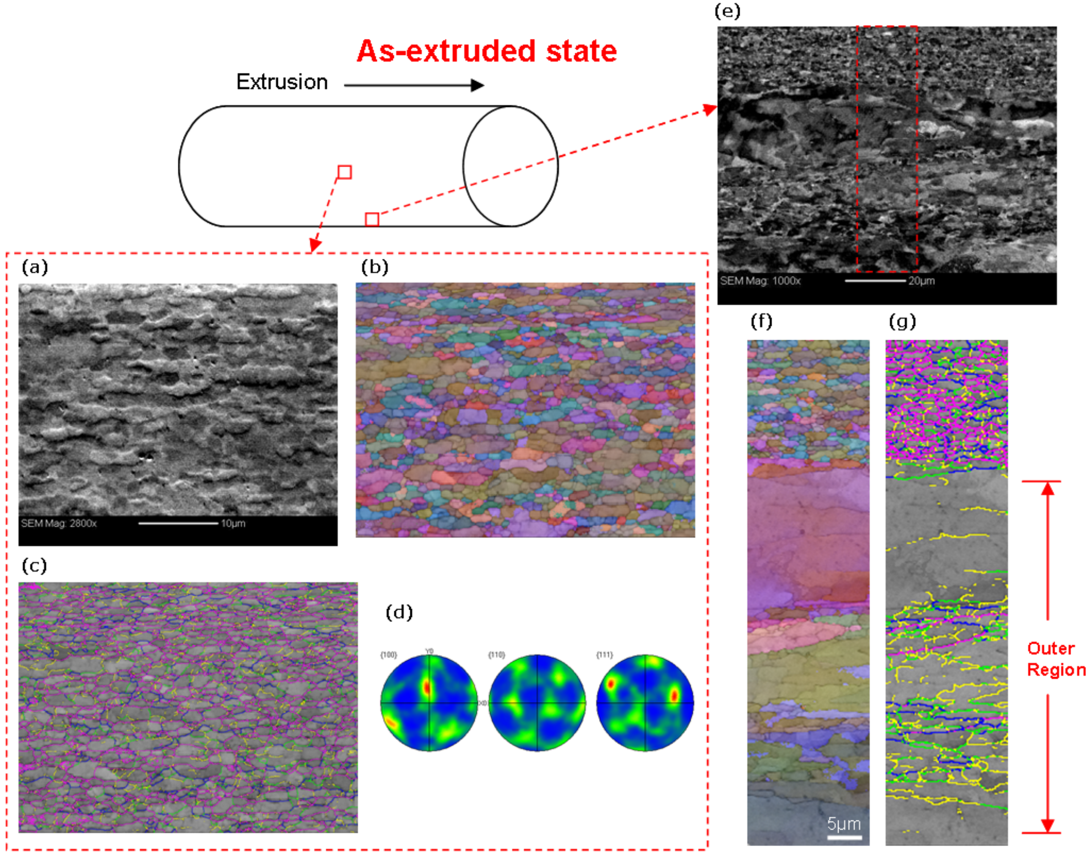
d) Deformed + SR
1380^{0}C/1h
Transverse section. SEM.





e) Deformed + SR
1380^{0}C/1h
Plan section. SEM.

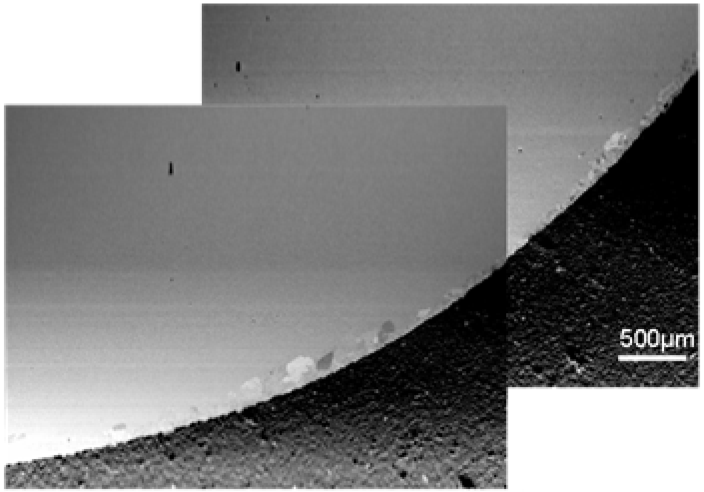
Recrystallisation of extruded PM2000 bar



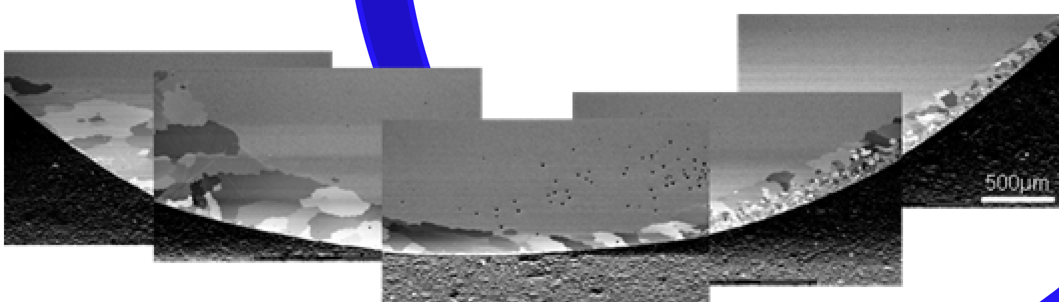
Predominance of labg's near bar surface

PM2000 bar (can/HIP/hot extrude (1000^0C). Fine-grained as-extruded state. SEM longitudinal sections

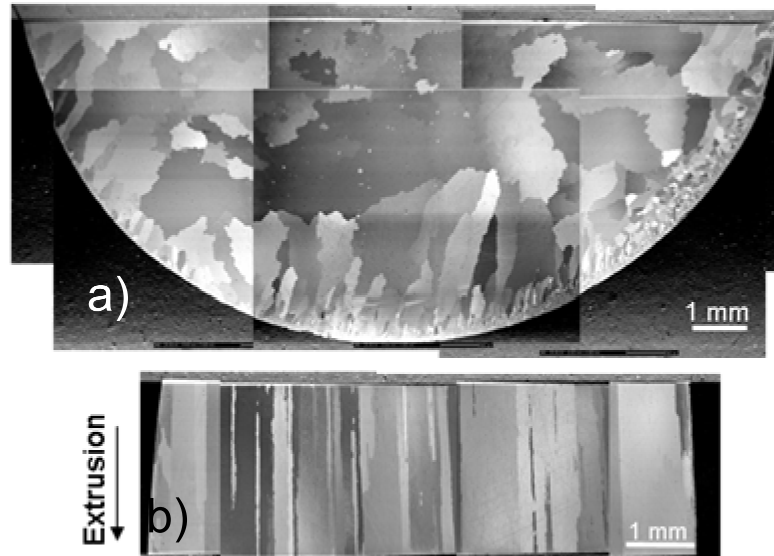
Recrystallisation of extruded PM2000 bar



As–extruded + 900⁰C/1h. SEM transverse section

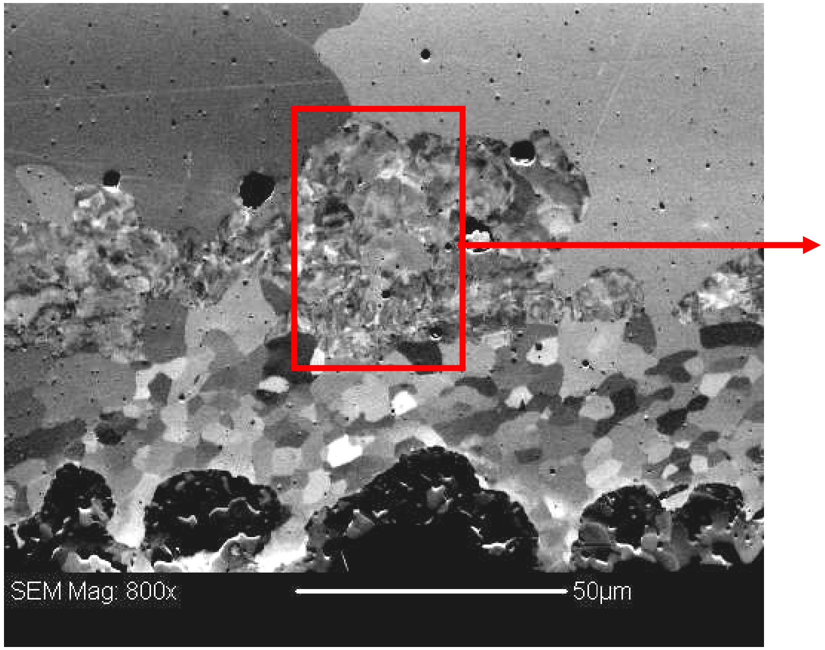


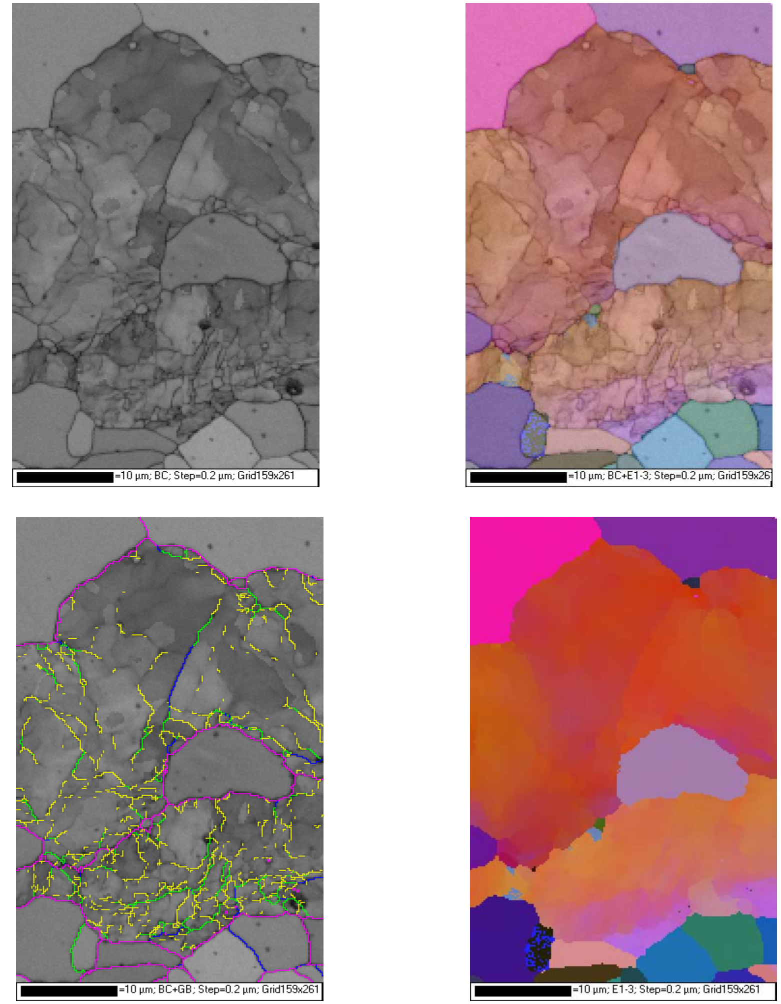
As–extruded + 1100⁰C/1h. SEM transverse section



As–extruded + 1200⁰C/1h.
a) SEM transverse section
b) SEM longitudinal section

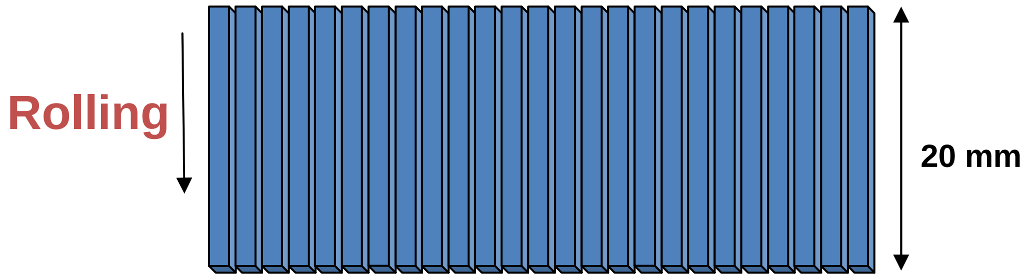
Recrystallisation of extruded PM2000 bar



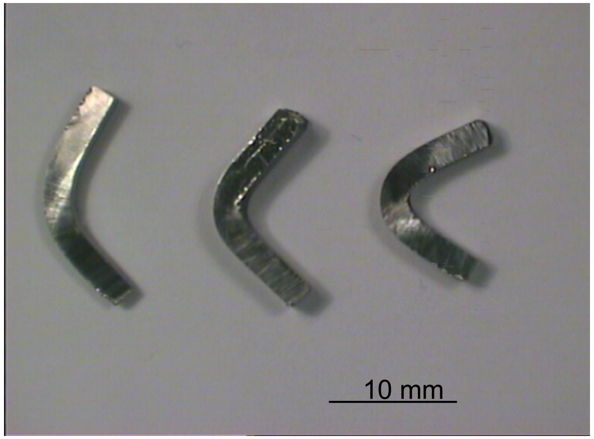


As–extruded + $1380^0C/1h$.
Transverse section. SEM.

PM2000 sheet subject to bending/twisting +SR

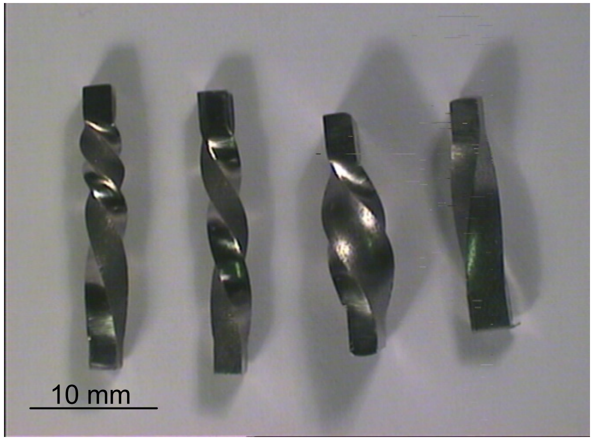


Bent samples



120º → 90º → 45º

Twisted samples



360º →270º → 180º → 90º

Samples deformed (bend/twist then SR (1380^{0}C /1h).

PM2000 sheet subject to bending/twisting +SR

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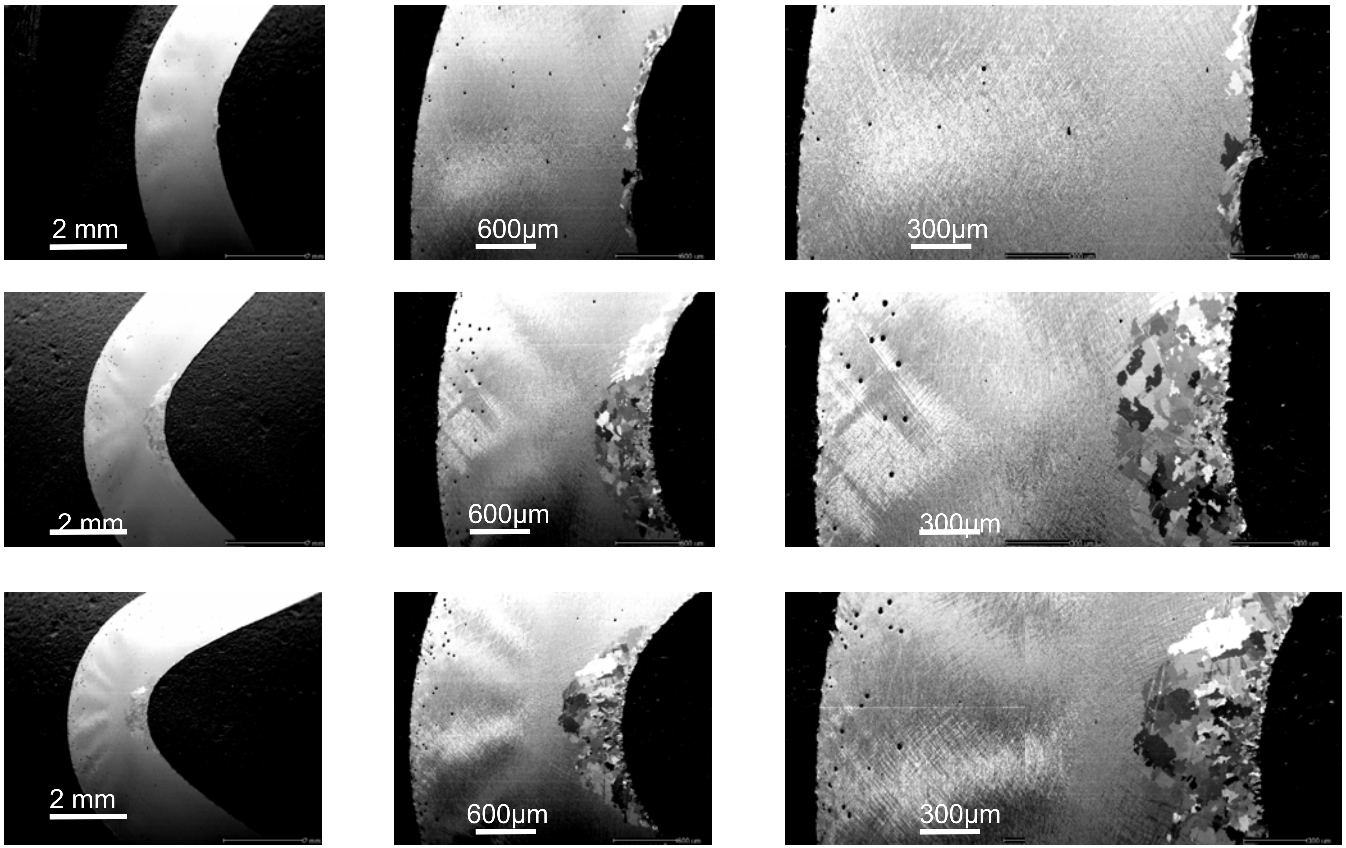
Final angle of bend

120^0

90^0

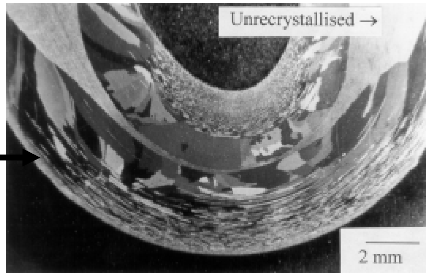
45^0



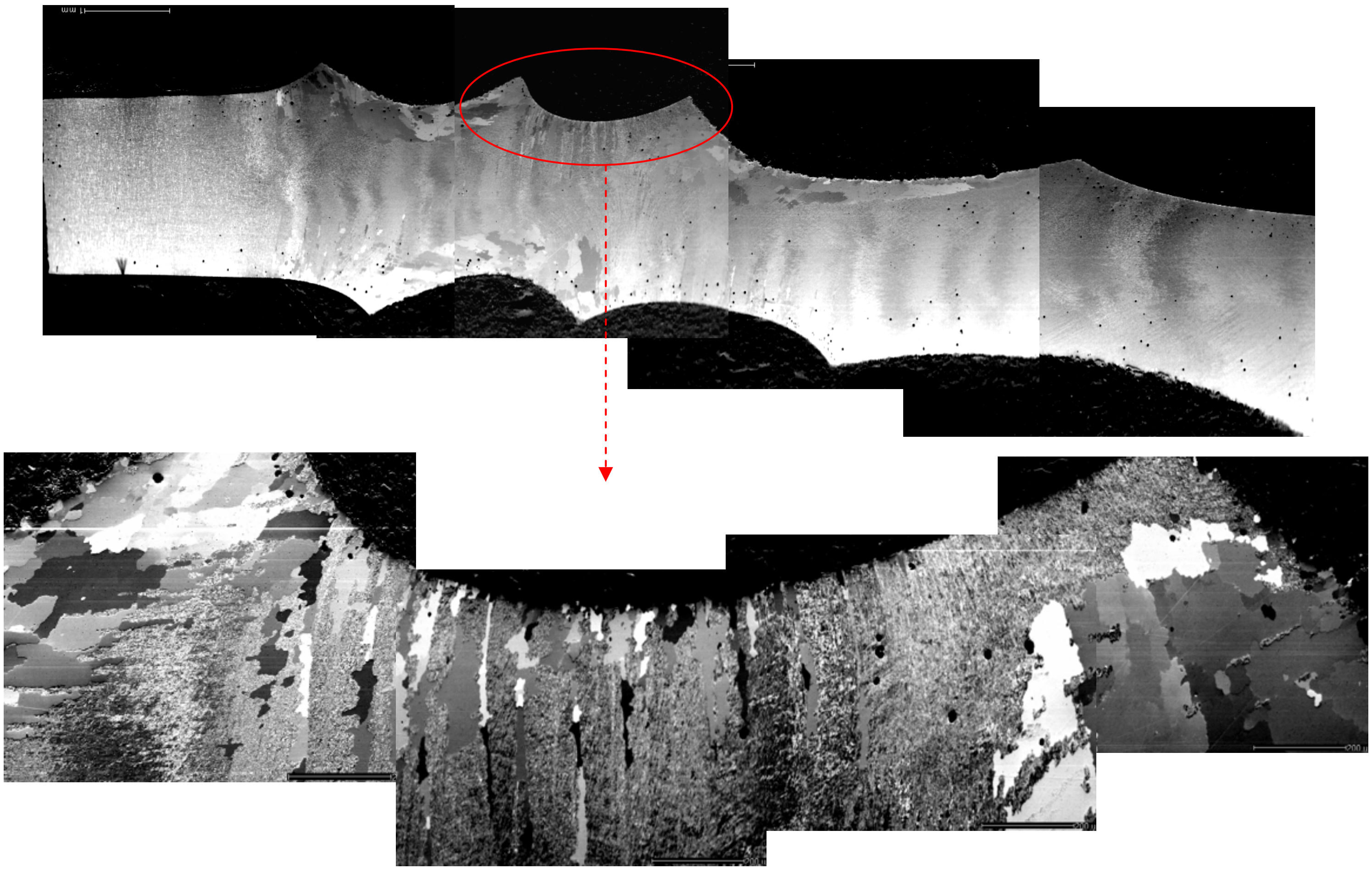
Fine grained samples deformed in bending + SR (1380^0C /1h).

MA957/☐-section from plate/ 30^0 final bend angle + 1300^0C/1h

(after Capdevila and Bhadeshia, 2000)



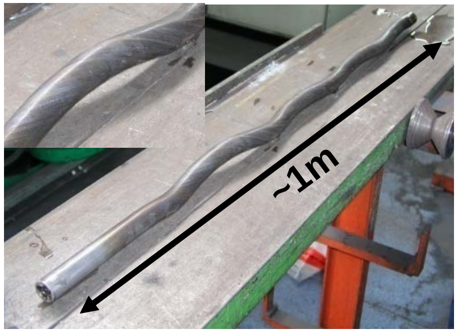
PM2000 sheet subject to bending/twisting +SR



PM2000 ‘bar’ sample cut from sheet, twisted 360^0 + SR (1380^0C /1h).

Torsional forming trials: ASTM 446 steel tube

- Torsion trials: Forecreu, France → Kennametal Inc, Evans GA (twist drill manuf.)



- Kennametal trials unfunded; tube supplied free issue ex-UoL
- PM2000 replaced by Sandvik 4C54 (ASTM 446) heat resisting seamless Cr ferritic steel tube

Sandvik 4C54 chemical composition wt.%

C max	Si	Mn	P max	S max	Cr	N	Fe
0.2	0.5	0.8	0.03	0.015	26.5	0.2	Balance

- tube 21.3mm diameter, 2.7mm wall; arrangement supported by internal steel mandrel

Torsional forming trials: ASTM 446 steel tube

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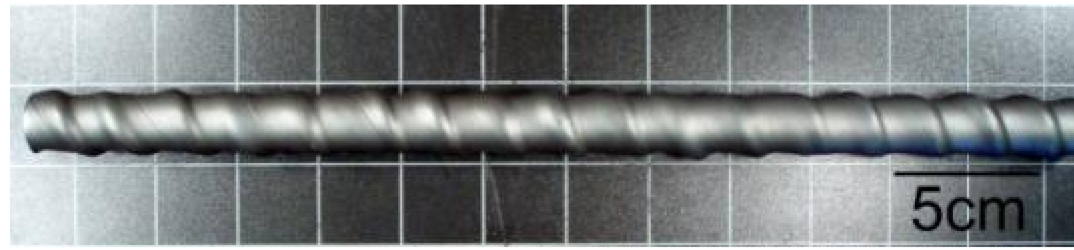
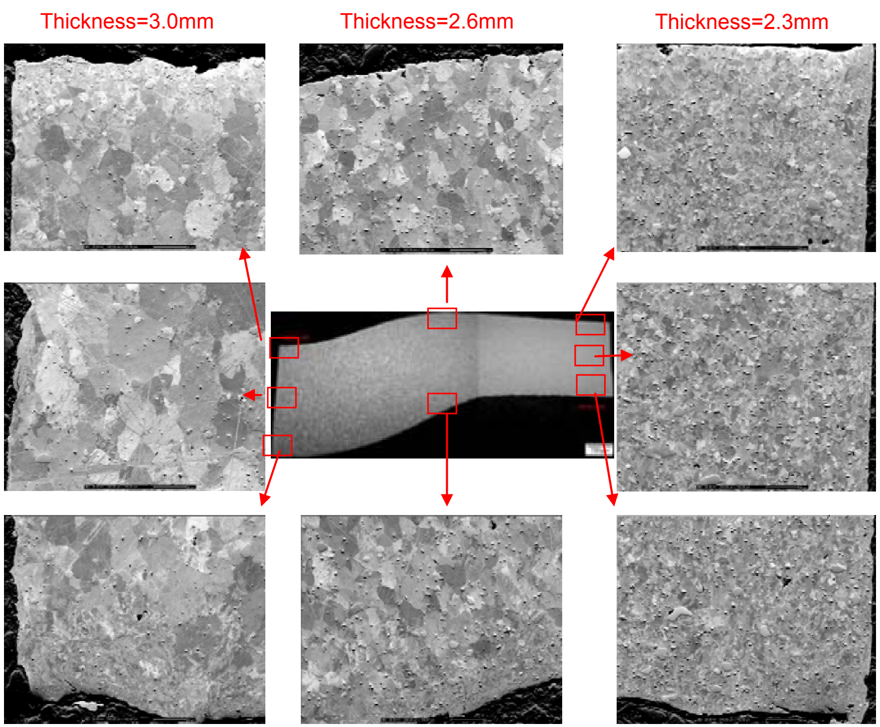
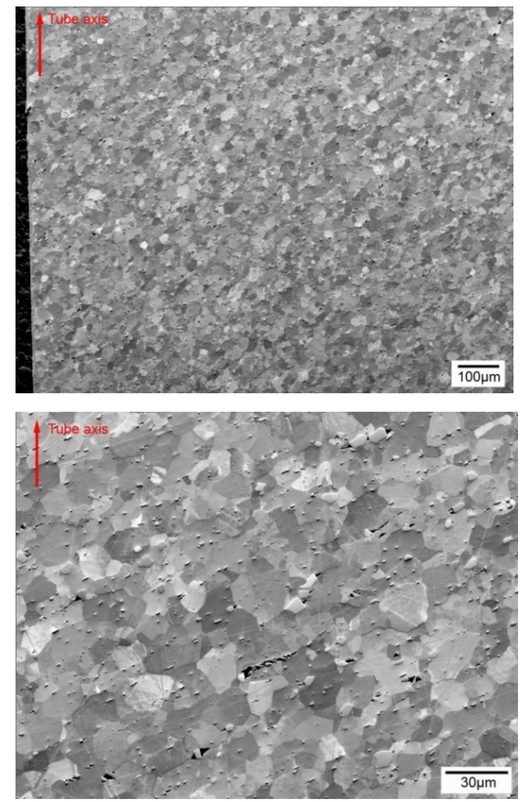
- Sandvik 4C54 (ASTM 446) tube +
  internal steel mandrel (15.5mm diam.)
- Initial parameters:
  torsion at ~1000^0C/ 0.55 r.p.m/ mm.s^{-1} hot zone
- outcomes:
  60^0 torsion achieved/wall thinning+thickening/
  tube dynamic recrystallisation





Longitudinal section through a 'ridge'. SEM channelling contrast. Original tube wall thickness 2.7mm.



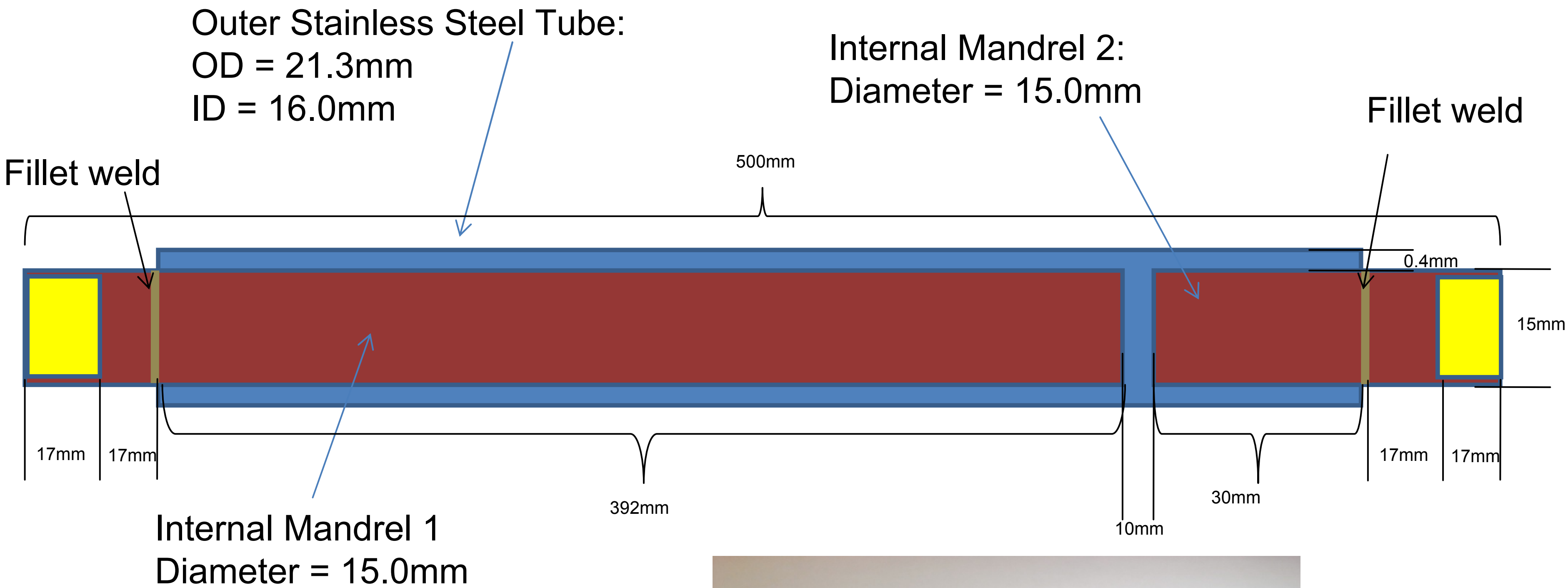
Transverse section showing 60º inclination of stringers in microstructure.

Torsional forming trials: ASTM 446 steel tube

Trial 2:

- Fixed mandrel
- 950^0C
- 0.55 r.p.m
- 7mm traverse speed
- 60^0helix

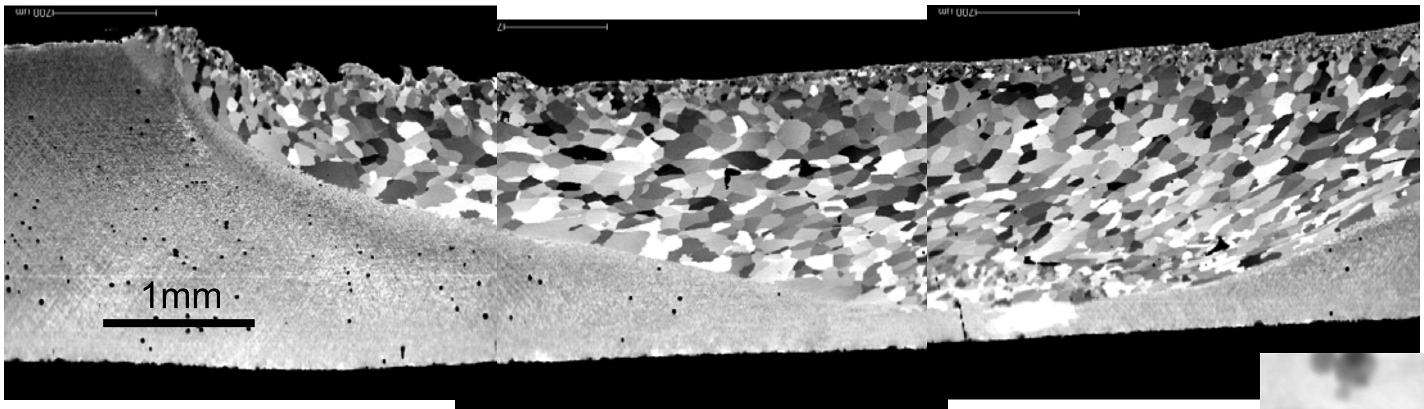
Torsional forming trials: ASTM 446 steel tube



Trial 5:

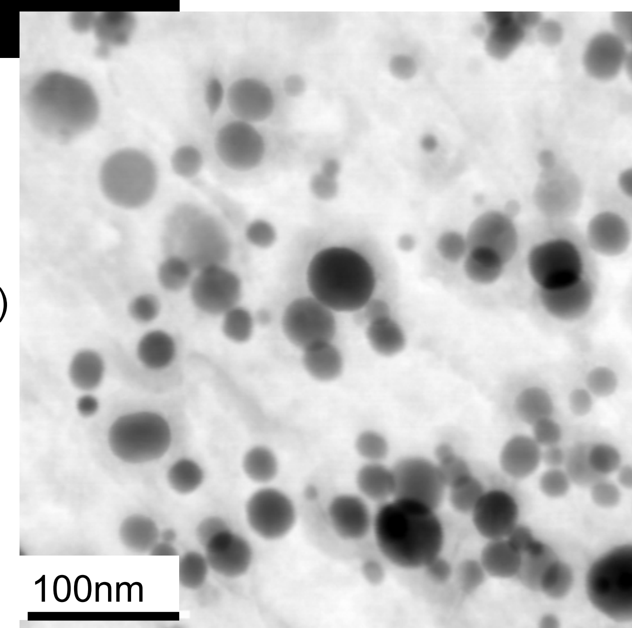
- Split mandrel
- < 900^0C
- 0.3 r.p.m
- 7.2 mm traverse speed
- ~30^0helix

Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation



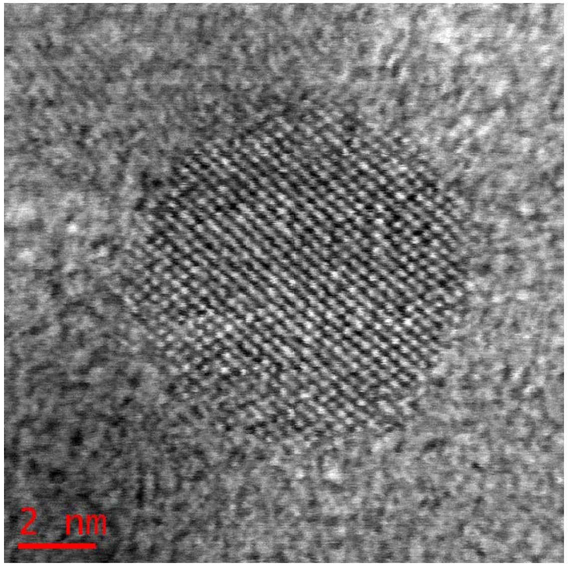
Cross section of friction stir weld in PM2000 sheet + SR 1380^{0}C/1h

- PM2000 sheet in fine-grained (KKL4) condition + friction stir weld (TWI)
- FS joint + SR 1380^{0}C/1h
- carbon extraction replicas from parent sheet and joint

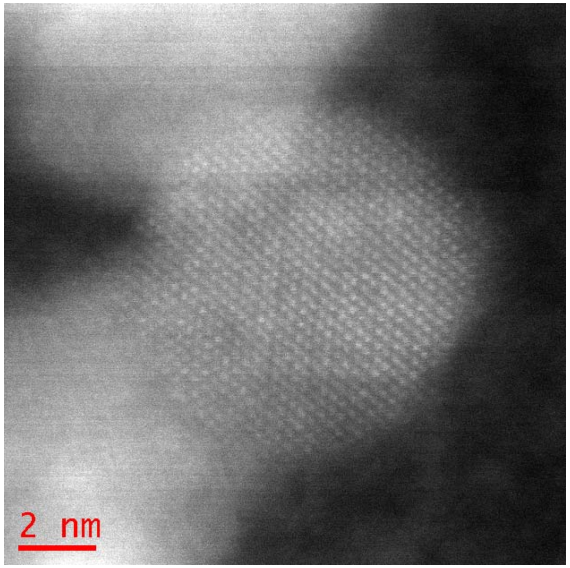


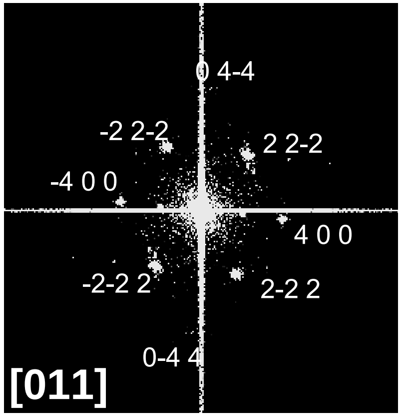
Carbon extraction replica of sample

Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation



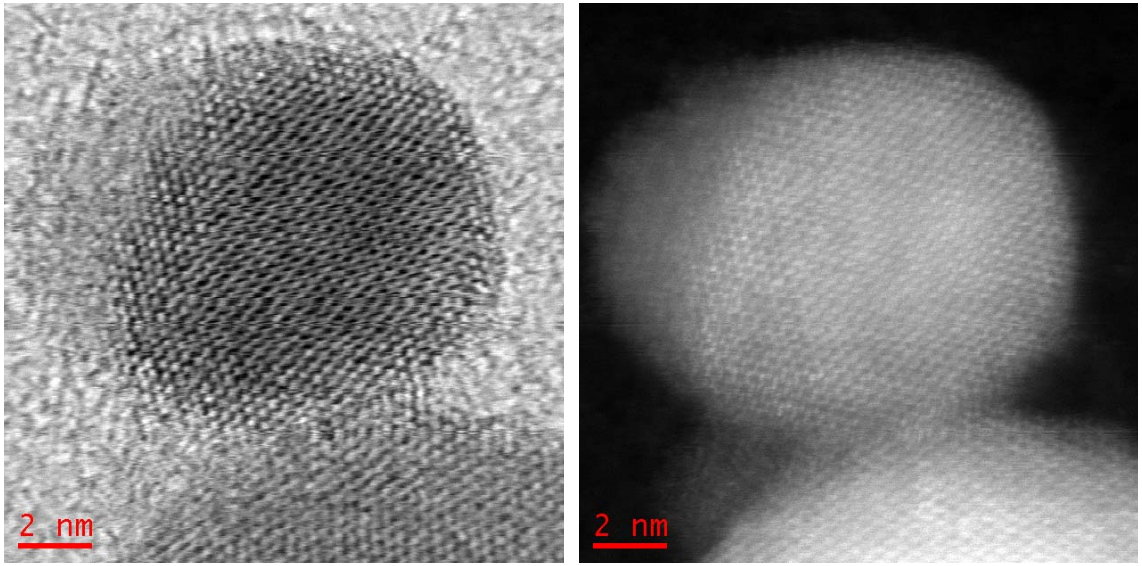
Cubic -Y_2O_3





Extraction replica from friction stir weld in PM2000 sheet + SR 1380^{0}C/1h

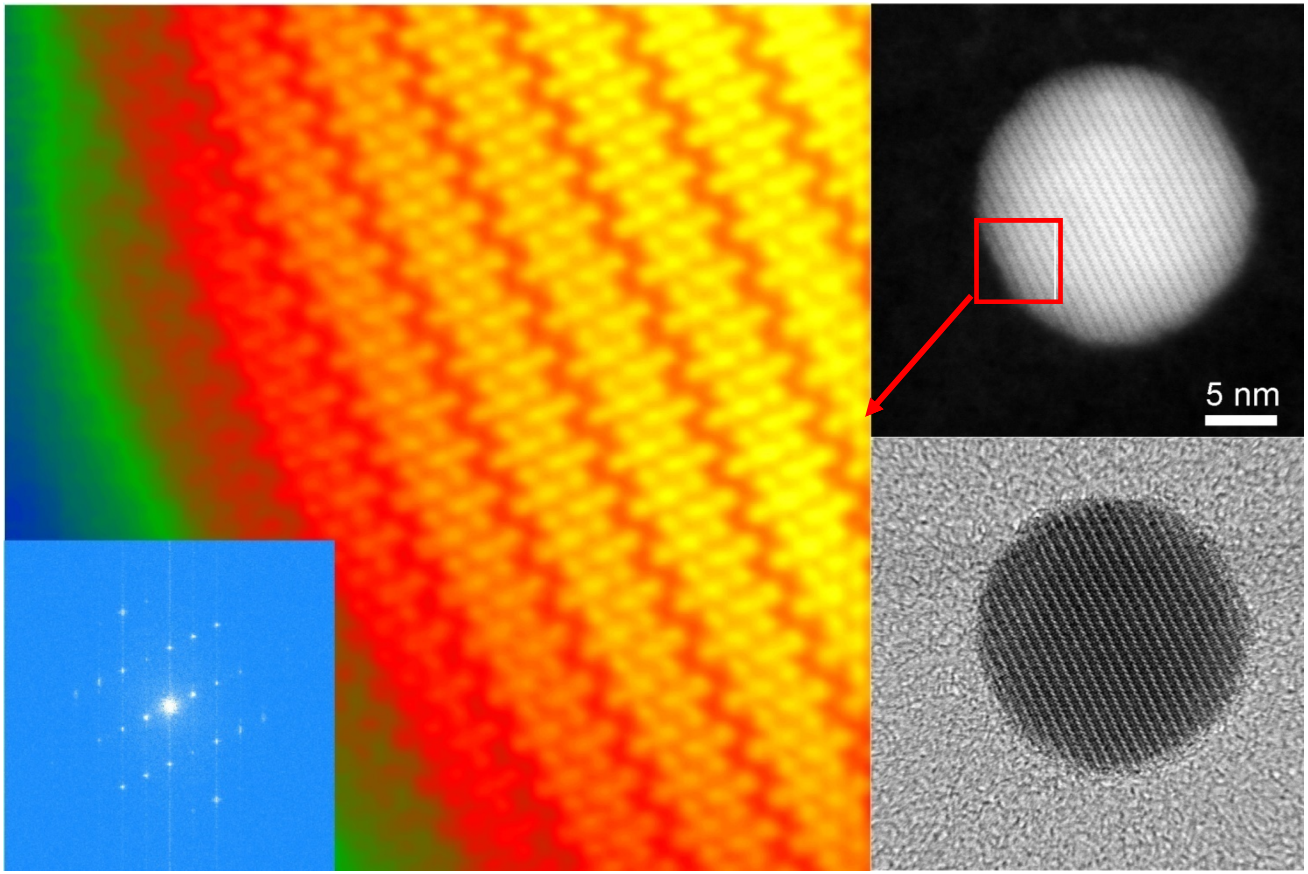
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation



$Y_3Al_5O_{12}$ Yttrium-Aluminium-Garnet (YAG)

Extraction replica from friction stir weld in PM2000 sheet + SR 1380^0C/1h

Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation



$YAlO_3$ Yttrium-Aluminium-Perovskite (YAP)

Extraction replica from friction stir weld in PM2000 sheet + SR 1380^{0}C/1h

Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

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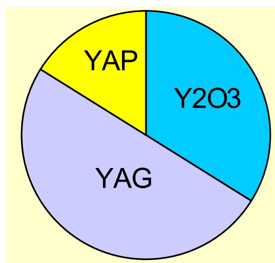
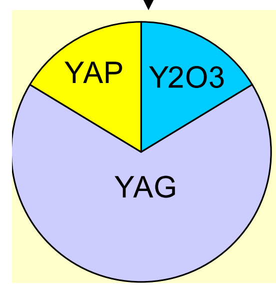
Friction Stir Weld

As-received Alloys



Y_2O_3 - 34%
YAG - 50%
YAP - 16%

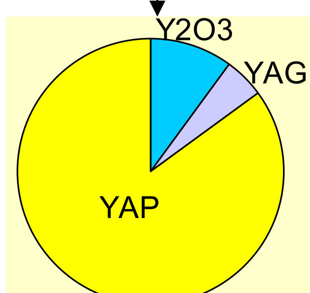


Y_2O_3 - 16%
YAG - 66%
YAP - 16%

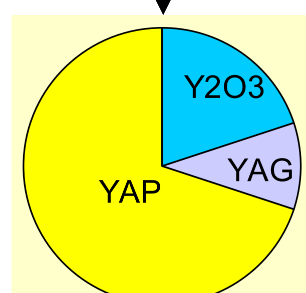
Y_2O_3 or YAG
⇩
YAP

Recrystallization Treatment (1380ºC, 1hr)

Y_2O_3 or YAG
⇩
YAP



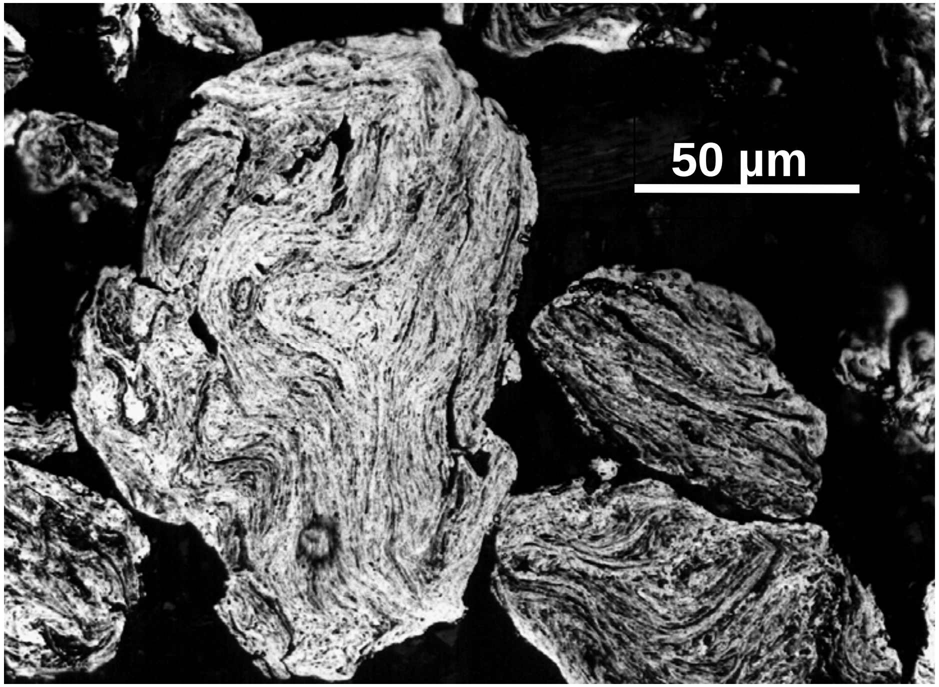
Y_2O_3 - 10%
YAG - 5%
YAP - 85%



Y_2O_3 - 20%
YAG - 70%
YAP - 10%

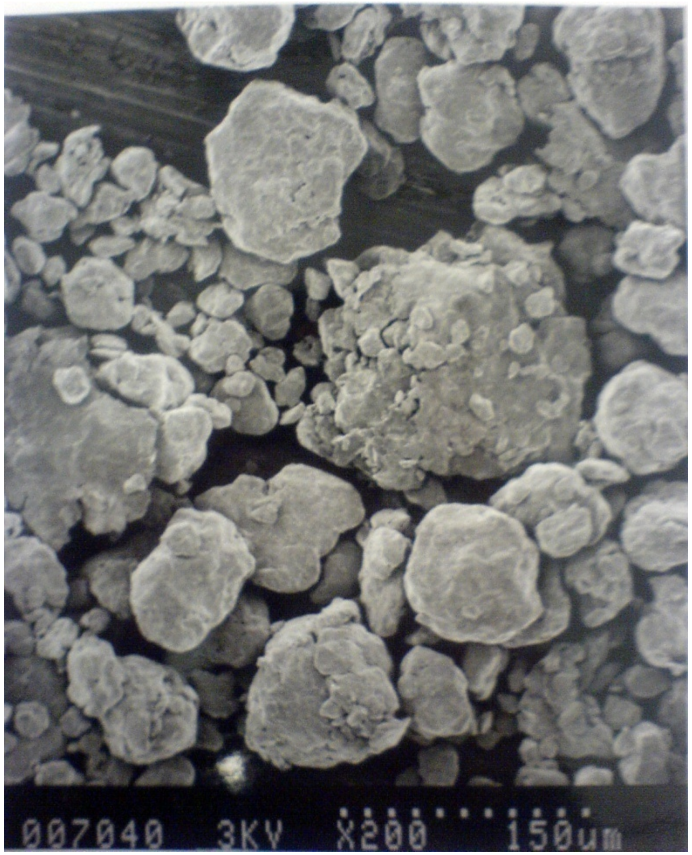
Replacement ODM751

ODM 751 powder – Dour Metal SA, Belgium



ODM751 MA powder particles, Dour Metal SA.

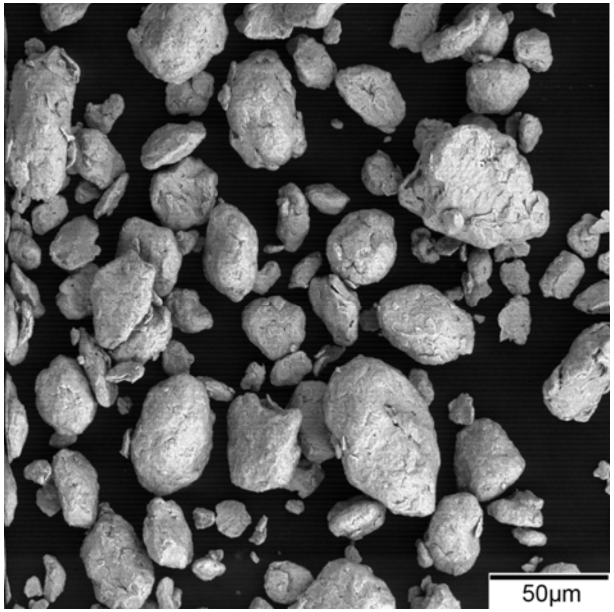
Optical micrograph, etched transverse section.



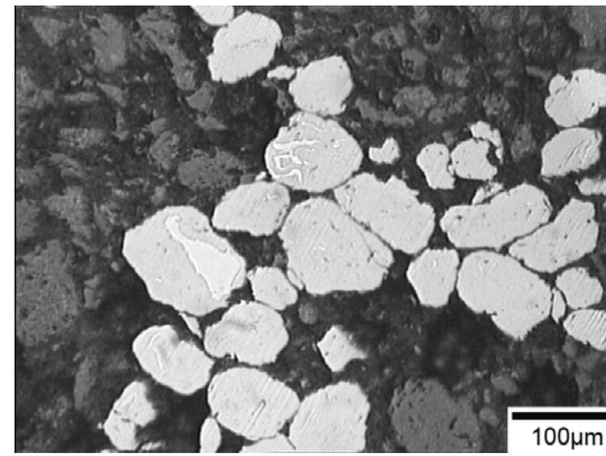
ODM751 MA powder. SEM image un-sieved powde

Replacement ODM751

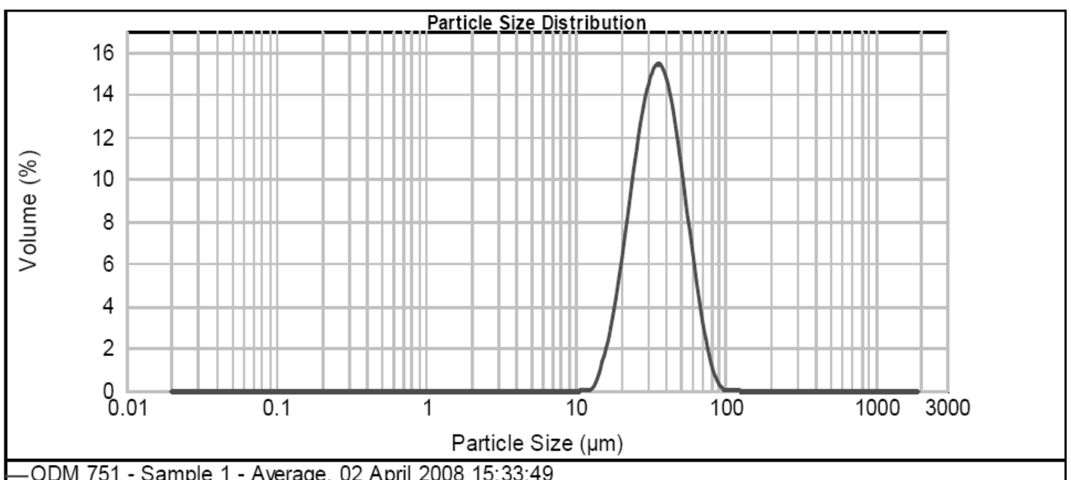
- Powder supplied by **Dour Metal, s.r.o.**
- Powder sample dispersed in H_2O/surfactant.
- Laser diffraction measurements (5/sample).
- Results averaged over five tests.
- Powder size range: ~10-110µm.
- Mean size: ~35 µm.

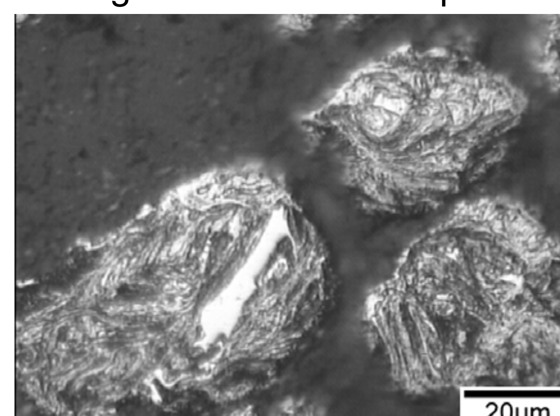


Replacement ODM751 sample. Backscattered SEM image



Polishing relief: un-etched optical sectio





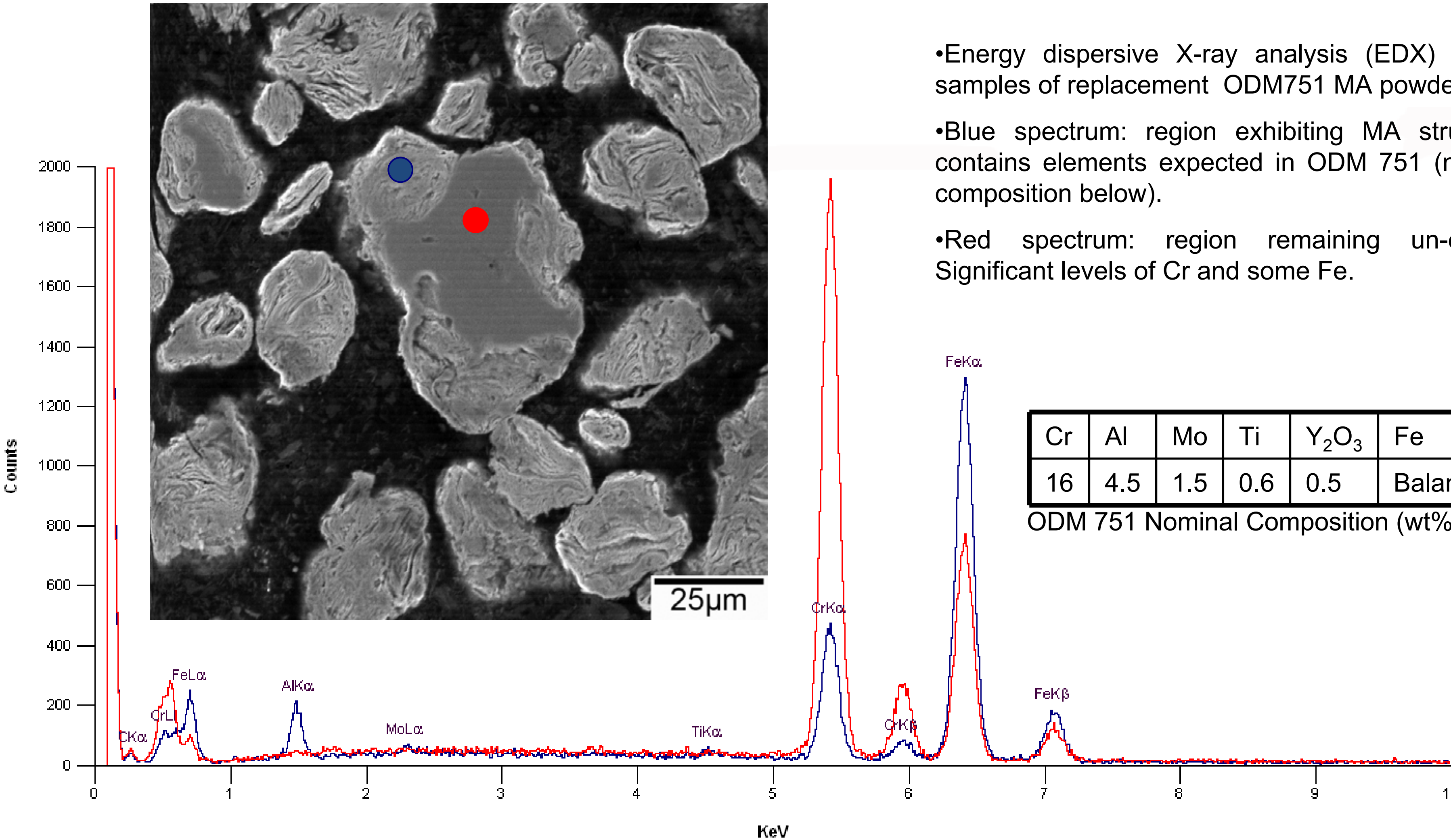
Etched sample: optical section

Replacement ODM751

•Energy dispersive X-ray analysis (EDX) of trial samples of replacement ODM751 MA powders.

•Blue spectrum: region exhibiting MA structures contains elements expected in ODM 751 (nominal composition below).

•Red spectrum: region remaining un-etched. Significant levels of Cr and some Fe.



Cr	Al	Mo	Ti	Y_2O_3	Fe
16	4.5	1.5	0.6	0.5	Balance

ODM 751 Nominal Composition (wt%)

Replacement ODM751

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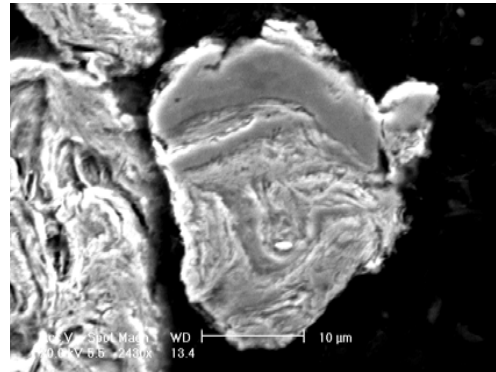
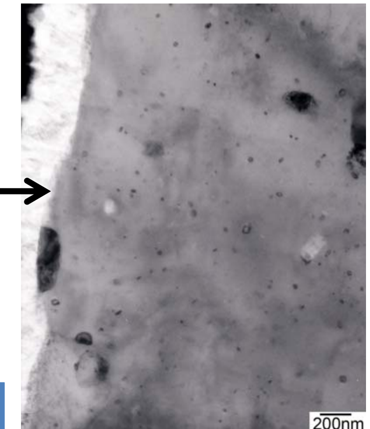
- EDX measurements from trial sample of replacement ODM751 MA powder (as shown in SEM image of polished/etched sample).
- Tabulated values normalised without the carbon contribution.

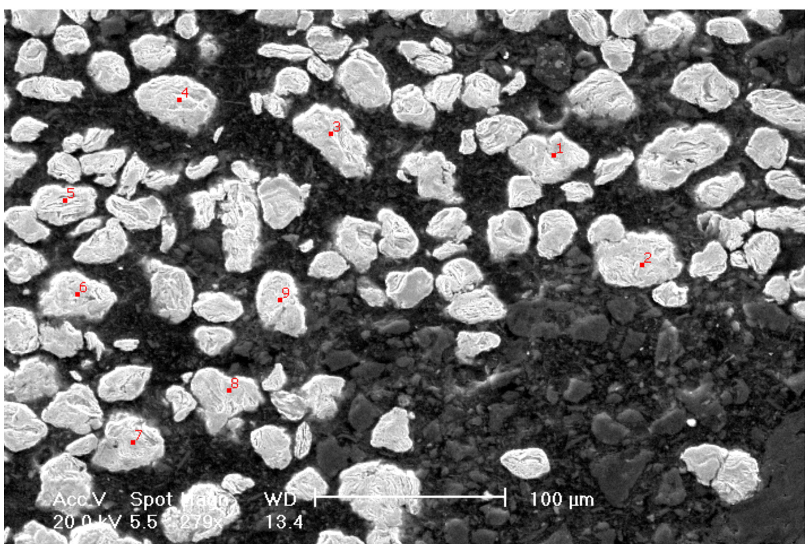




ODM751 powder
+900^{0}C/1h.
TEM BF, FIB sampl

Element	Weight %	
	Sample	ODM751 (nominal)
Cr	16.4 ± 1.6	16
Al	4.8 ± 0.8	4.5
Mo	1.6 ± 0.1	1.5
Ti	0.6 ± 0.1	0.6
O	3.4 ± 0.9	
Y_2O_3		0.5
Fe	73.2 ± 1.4	bal

Error bounds are one standard deviation

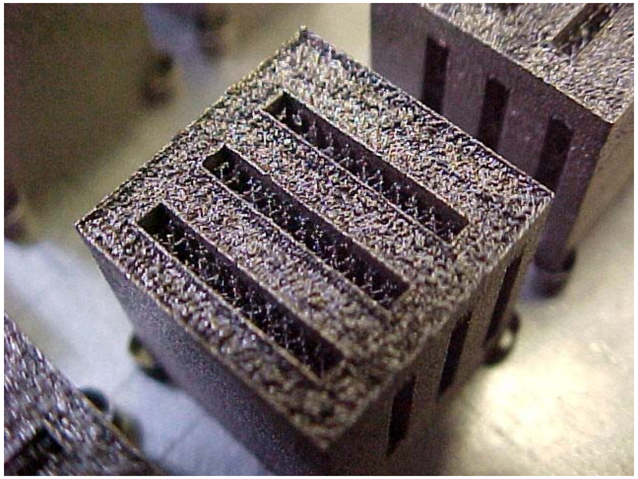


Replacement ODM751 MA powder. SEM image

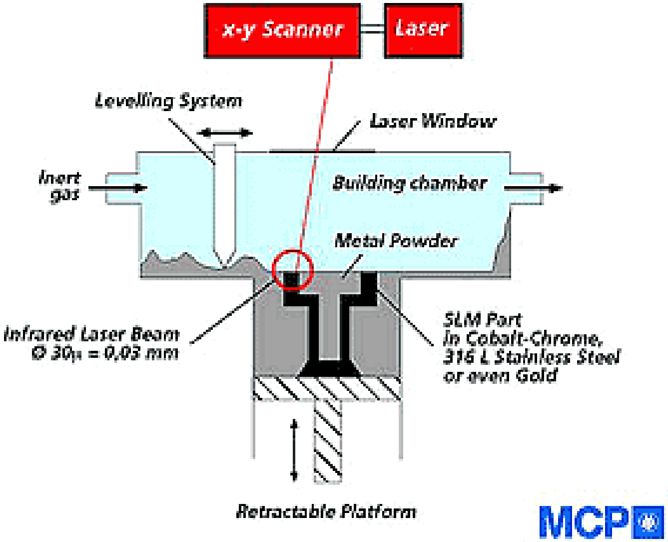
Selective Laser Melting (SLM) of PM2000 alloy powder

PM 2000 Thermocouple Sleeves
Application: e.g. in Gas Turbines; temperature measurement close to combustion chamber

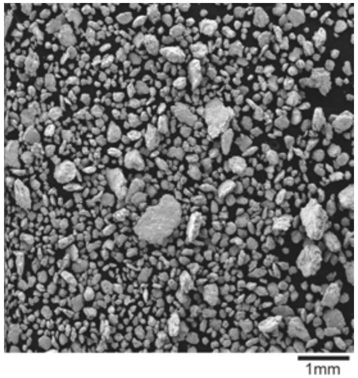
PM 2000 Burner Nozzles
Fuel injection in diesel engines (test parts)

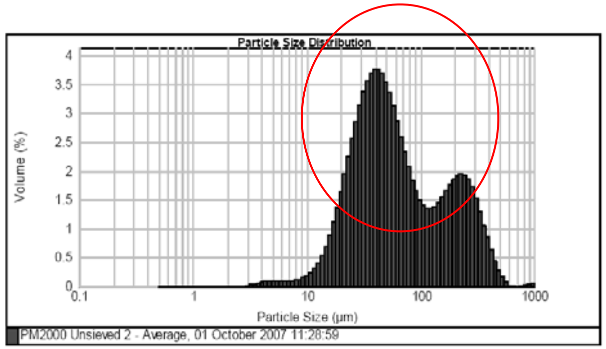


Micro-heat exchanger by SLM (20mm cube)

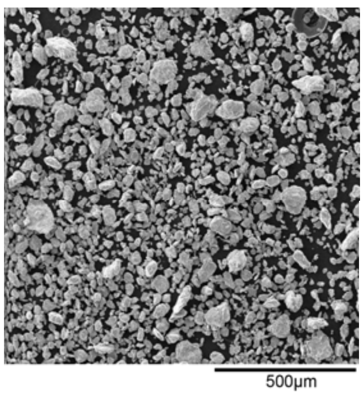


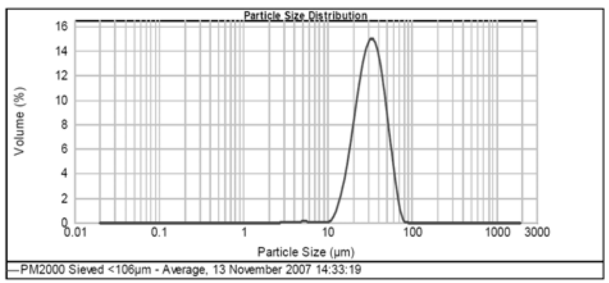
Selective Laser Melting (SLM) of PM2000 alloy powder



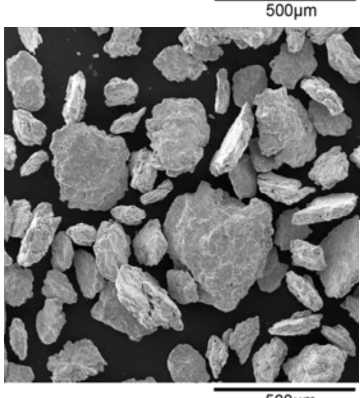


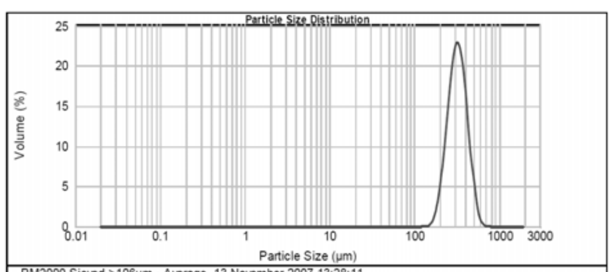
As-received powder





Smaller fraction <106µm





Larger fraction >106µm

PM2000 Particle size analysis

- The PM2000 MA powder exhibited a bi-modal particle size distribution.
- Powders were sieved to remove large particles that would impede flow/disturb the powder bed.

Selective Laser Melting (SLM) of PM2000 alloy powder

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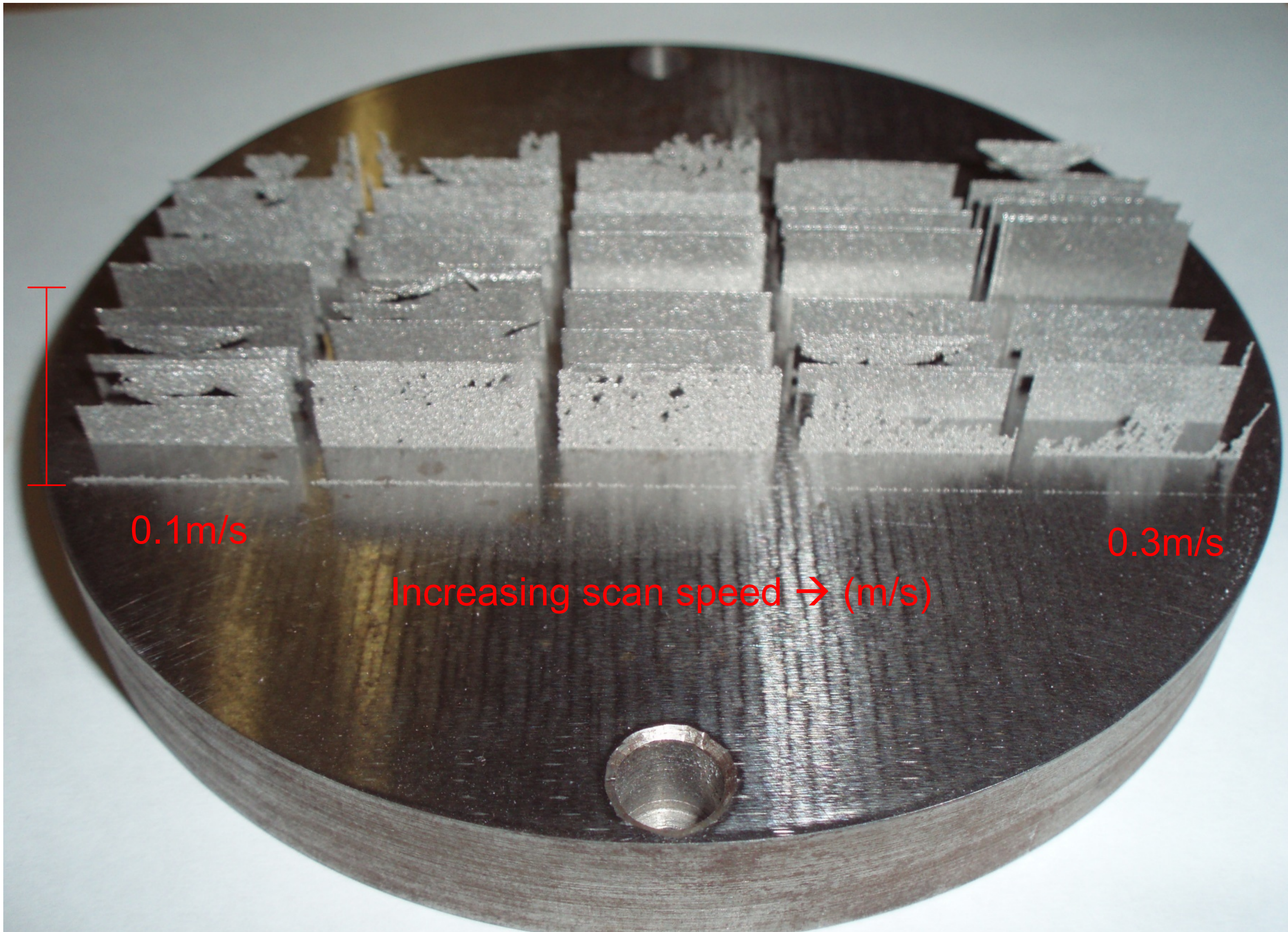
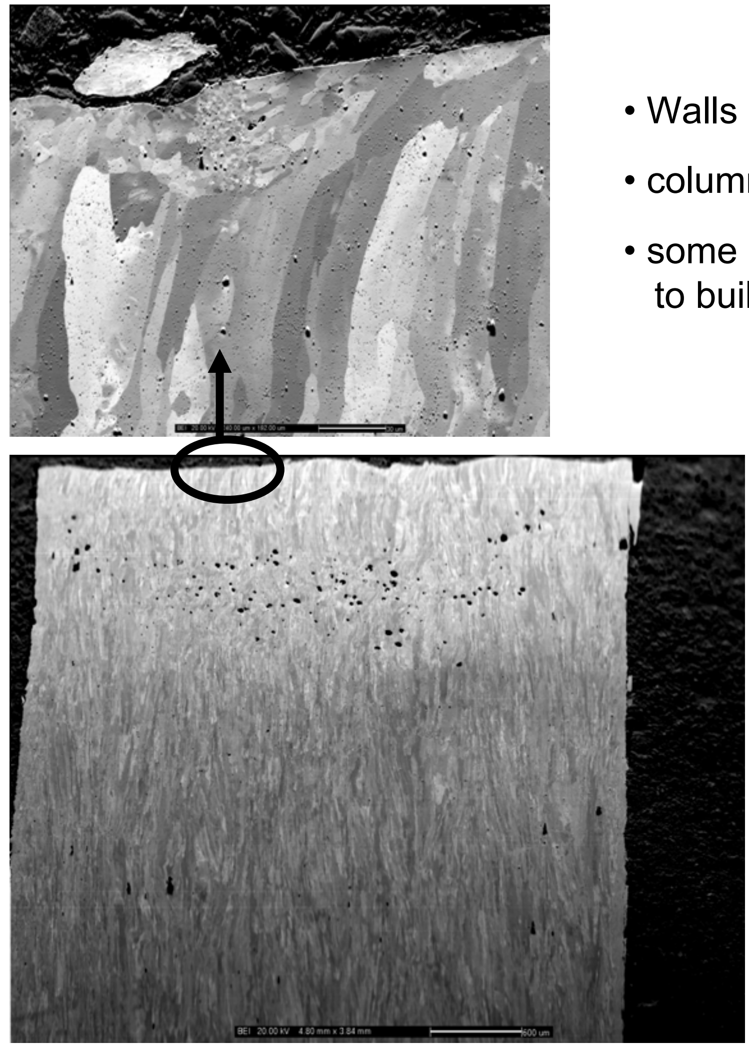


SLM of sieved PM2000 powder using MCP Realiser 100 unit. Ar-atmosphere. Mild steel base plate.

Selective Laser Melting (SLM) of PM2000 alloy powder

50W, 0.1m/s wall

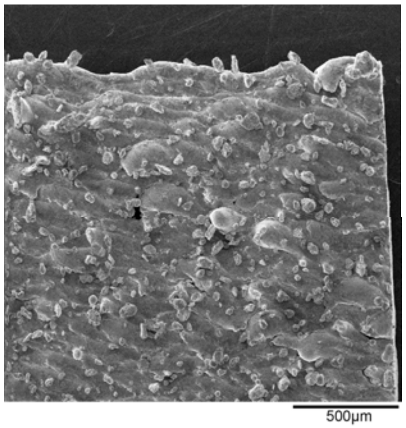


PM2000 SLM wall, side view, 50w, 0.1m/s.
Electron channelling contrast images.

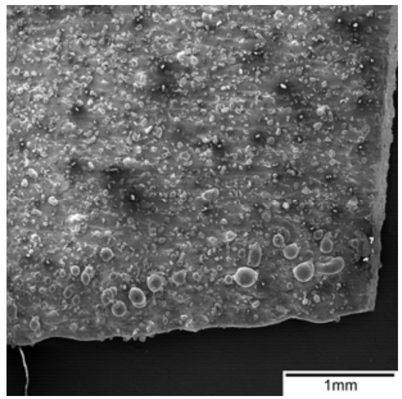
- Walls ≤ 200μm thickness
- columnar grain structure
- some porosity; sensitive to build parameters

Selective Laser Melting (SLM) of PM2000 alloy powder

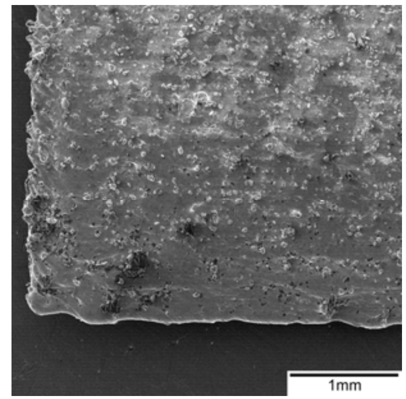
Wall integrity as a function of laser power (W) and scan speed ($m.s^{-1}$)



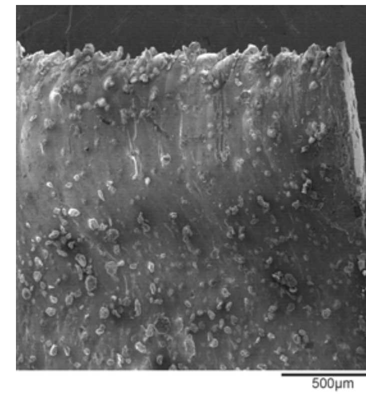
20w, 0.1$m.s^{-1}$



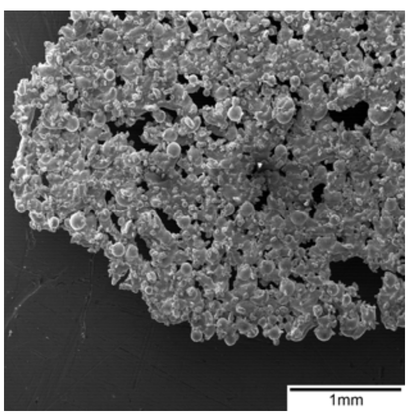
30w, 0.1$m.s^{-1}$



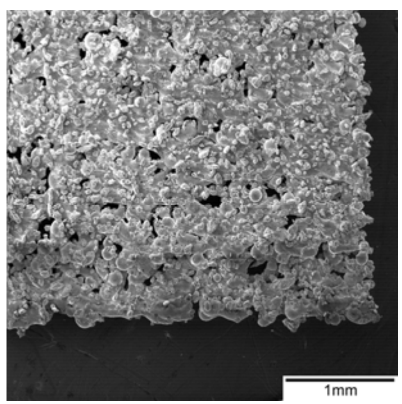
40w, 0.1$m.s^{-1}$



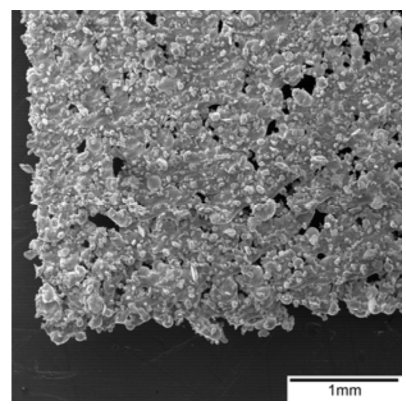
50w, 0.1$m.s^{-1}$



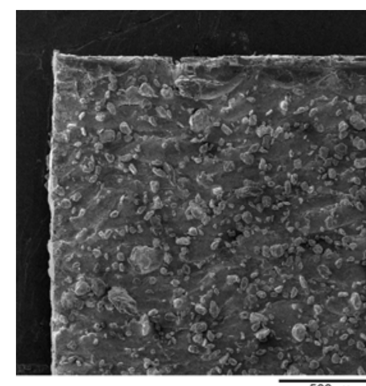
20w, 0.15$m.s^{-1}$



30w, 0. 25$m.s^{-1}$



40w, 0.3$m.s^{-1}$



50w, 0.3$m.s^{-1}$

Selective Laser Melting (SLM) of PM2000 alloy powder

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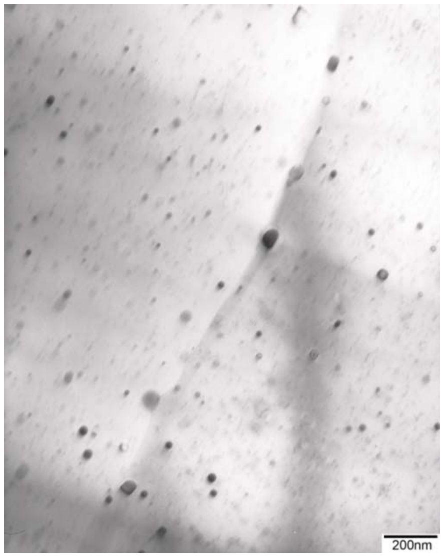
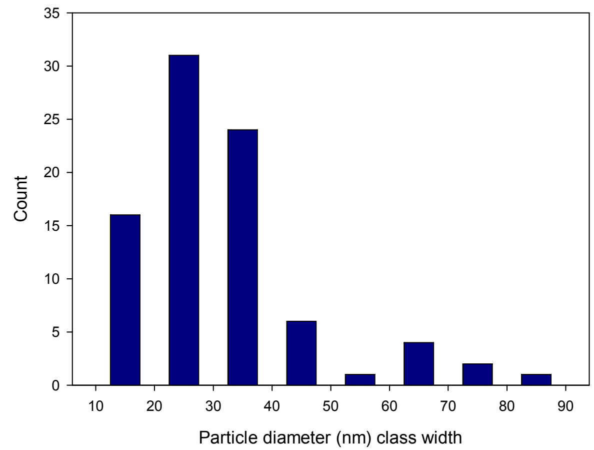
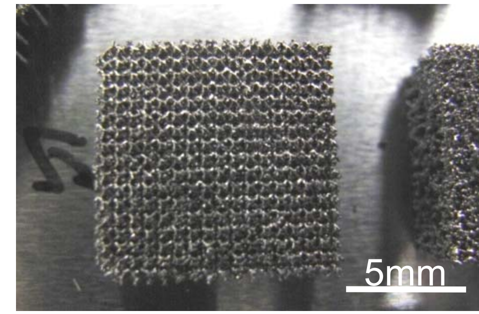


PM2000 tube (SR 1380^{0}C/1h)

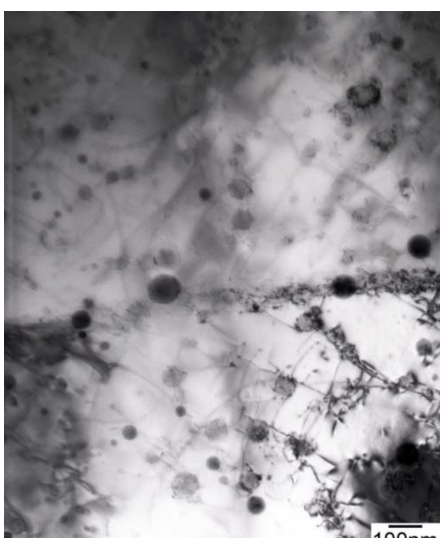
Nominal oxide particle diameter = **30.8 ± 15.1nm**



ODS particle diameter - PM2000 twisted tube annealled for 60mins at 1380ºC

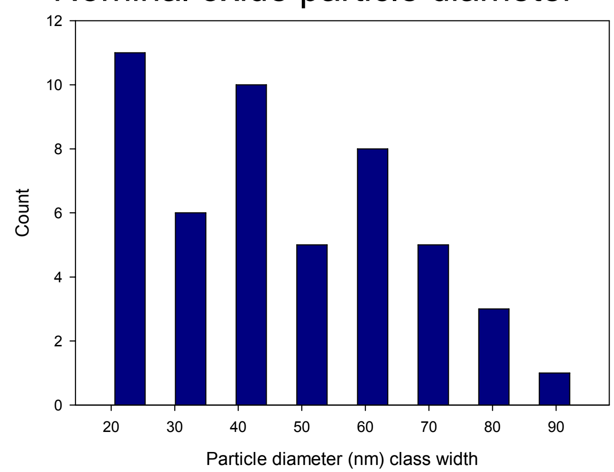


PM2000. SLM mesh build.
50W/ 0.2ms^{-1}.
0.6mm octahedral cell.



PM2000 SLM Wall, 50W, 0.2m.s^{-1}

Nominal oxide particle diameter = **45.8 ± 18.6nm**



ODS particle diameter - PM2000 SLM wall, 50W, 0.2m/s

Evidence of:

Retention of oxide particles in PM2000 after SLM fabrication process.

An increase in mean particle size following 50W/0.2m.s^{-1} SLM.

Summary

Recrystallisation of PM2000 tube, bar and sheet

(i) SR (1380^0C/1h) of torsionally deformed tube results in helical grain structures.
Grain size varies:

- across the wall
- around the circumference
- along the tube axis.

Isolated regions of PR material remain.

(ii) Grain size across SR product forms (tube, extruded bar and sheet samples subject to bending and torsion):

- Can be inhomogeneous
- Varies surface to bulk
- Is influenced by cumulative processing history

Torsional forming trial

(i) Torsion trials on ASTM 446 supported by Kennametal Inc

- 60^0 twist achieved
- Extensive plasticity/dynamic recrystallisation
- Tube ‘ribbing’ with local wall thickening/thinning
- Macroscopic straightness with fixed/split-mandrel

Summary

Evolution of oxide dispersions during secondary recrystallisation of PM2000

(i) Yttria/YAG/YAP present in sheet and friction stir weld before and after SR

(ii) YAG replaced by YAP as dominant oxide type during SR

Replacement ODM751

(i) Dour Metal, s.r.o. , Slovakia producing initial batches of replacement ODM751

(ii) Composition produced is close to legacy ODM751

(iii) Incomplete MA of residual amounts of Cr-rich precursor

SLM of PM2000 alloy powders

(i) Sieved PM2000 powders can be fabricated using SLM techniques

(ii) 'fully dense walls' can be built using a combination of laser power (W) and scan spe

(iii) Initial trials suggest ODS particles can be retained, though with some coarsening