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Electron backscatter diffraction as a tool for three dimensional orientation analysis

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6 IX 2012

• Interdisciplinary PhD Studies in Materials Engineering with English as the language of instruction •

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Conducted research

- 3D-EBSD measurements:
 - Ni/Mo protective coatings electrodeposited on steel substrate
 - Al6013 alloy after severe plastic deformation by the KOBO method
 - Crofer 22APU steel oxidized at elevated temperature
 - tetragonal ZrO_2 ceramics
- WDS measurements:
 - Crofer 22APU



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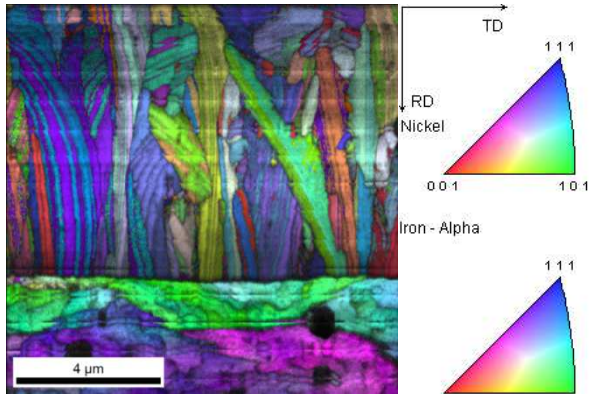


Ni-Mo/Fe

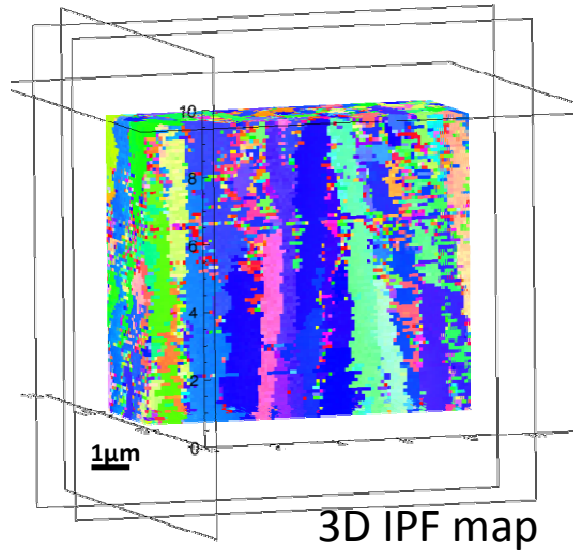
- Nickel based coatings obtained from environmentally friendly ammonia-citrate electrolytes are a promising alternative to chromium.
- Size, shape as well as orientation of Ni crystals containing Mo additives were established.



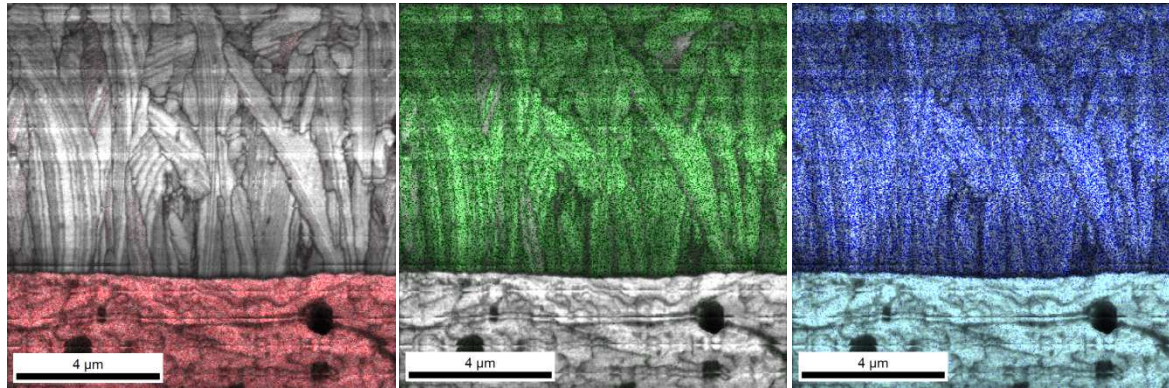
Ni-Mo/Fe results



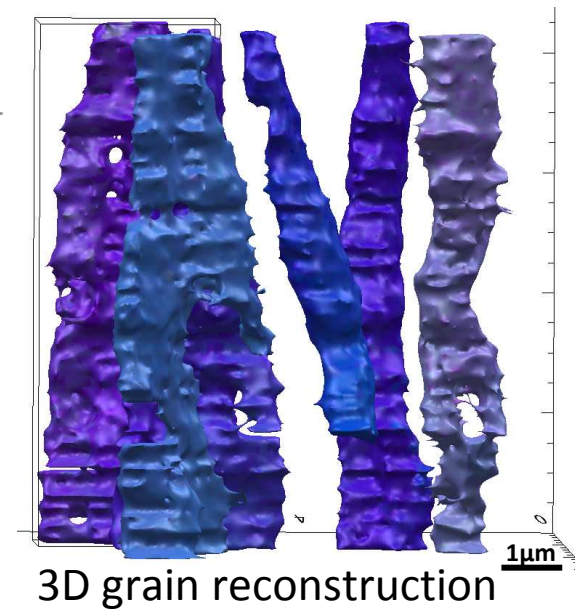
IPF map



3D IPF map



Fe Mo Ni
Chemical composition by EDS



3D grain reconstruction



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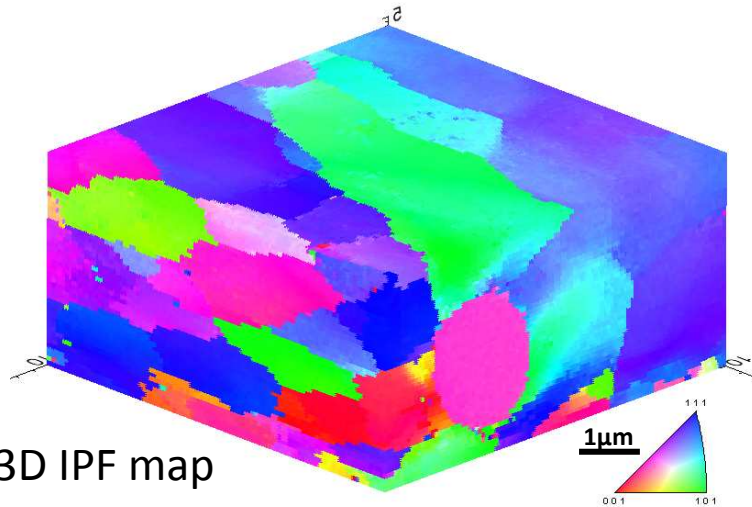


Al 6013

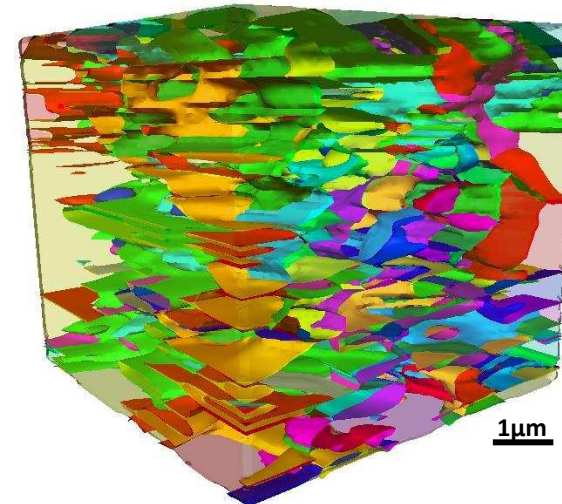
- Aluminum 6013 alloy was subject to complex deformation by the KOBO method which combines extrusion with simultaneous twist of the sample or dye.
- The resulting material possesses a gradient structure of the grain size distribution with fine crystallites in the outer part and elongated ones near the centre of the rod.



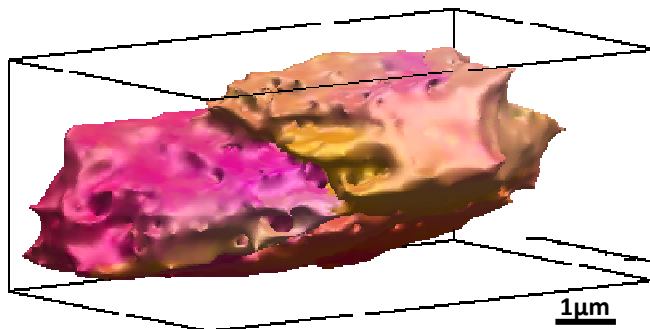
Al 6013 results



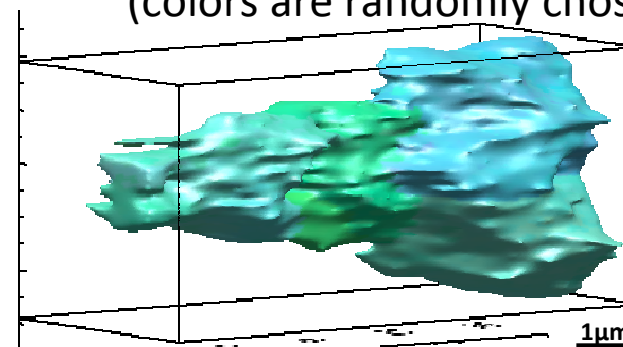
3D IPF map



grain boundary reconstruction
(colors are randomly chosen)



3D grain reconstruction



Pictures present 3D reconstructions of investigated samples. Colors denote crystallographic orientations unless otherwise stated. Color changes within crystallites in two lower pictures denote sub grain domains that may result from low angle boundaries.



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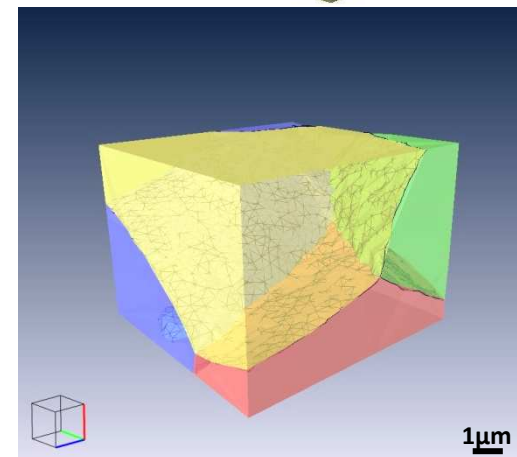
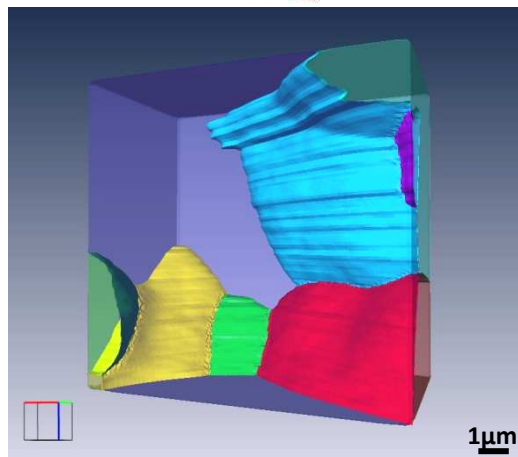
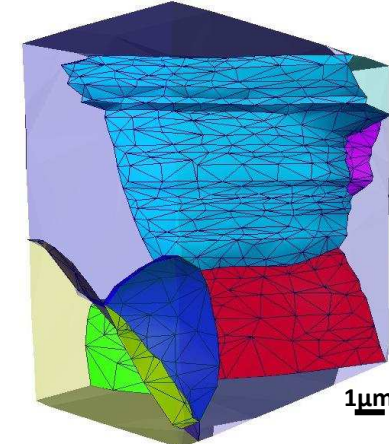
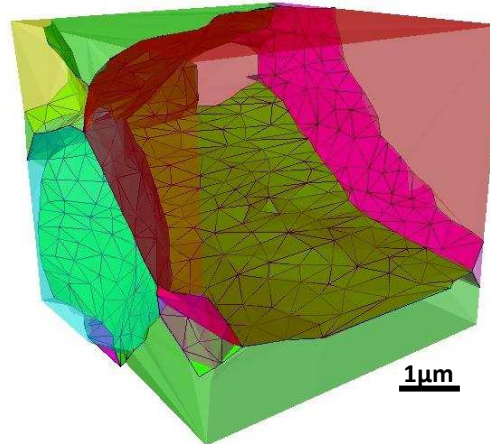
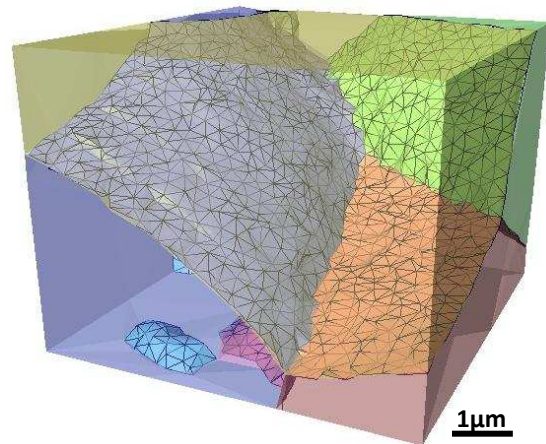


Crofer 22APU

- „Crofer 22APU” is a trade name of the steel developed for use in high temperature solid oxide fuel cells (SOFCs).
- It can be used as an interconnector material between cells and serves as electric current collector.
- When exposed to working conditions, a layer of chromium oxide (Cr_2O_3) is formed on the steel surface.
- 3D-EBSD measurements were carried out on steel substrate and an attempt to analyze oxide layers was done.
- The chemical composition of oxides was investigated using EDS and WDS to analyze the changes in concentration of steel additives like La, Ti, Cr and Mn.



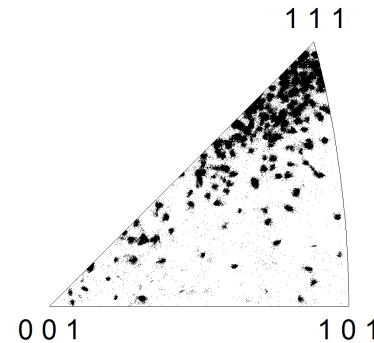
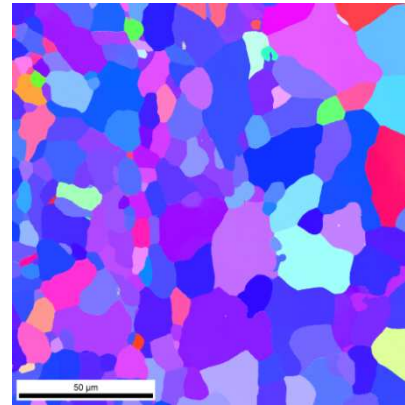
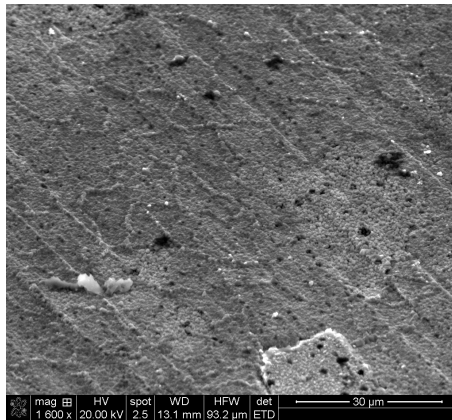
Crofer 22APU results



Pictures present 3D reconstructions of grain boundaries within steel samples. Colors are randomly chosen. In some pictures triangle meshes reconstructing grain boundaries are shown.



Crofer 22APU results



clean surface PF

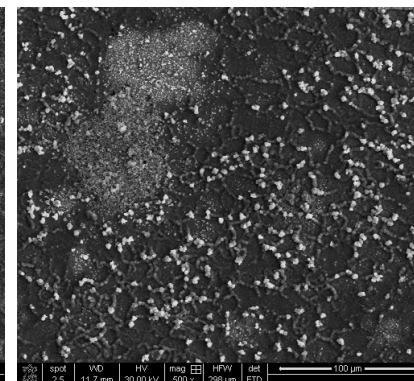
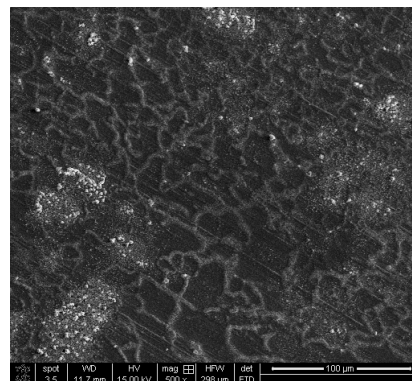
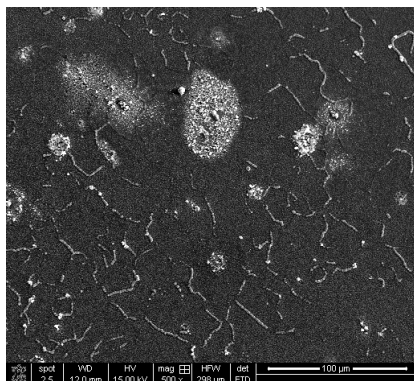
WDS	P1-24h	P2-48h	P3-72h
TiK	0,5	1,1	0,5
LaL	0,0	0,1	0,0
CrK	83,4	80,8	77,5
MnK	16,1	18,0	22,0

uncertainties to be established

clean surface IPF

FDS		Element	FeK	CrK	MnK	LaL	TiK	O K
steel			75,9±1,5	22,8±0,5	0,7±0,4	0,5±0,3	0,2±0,1	0,0
oxide layer	average		55,5±1,1	27,4±0,5	4,0±0,8	0,0	0,0	13,2±0,5
	crystallites		42,9±0,9	31,4±0,6	9,6±0,4	0,0	0,0	16,1±0,6
	flat area		58,6±1,2	28,9±0,6	0,6±0,3	0,0	0,0	11,9±0,5

Rows of Mn oxide crystallites probably represent the geometry of grain boundaries in samples. Analysis of chemical composition suggests that amount of Mn on the surface is increasing with time of oxidation.



Micrographs of oxide layers after 24, 48 and 72h oxidation.



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- The crystallographic structure of tetragonal zirconia polycrystals is quite close to cubic making it an affordable model material for 3D-EBSD investigations.
- Manufacturing of dense ZrO₂ ceramics is a well controlled process that enables production of sinters with required grain size and narrow size distribution.
- By compromising grain size with achievable investigated materials volume one can obtain satisfactory statistics for grain boundary characterization.



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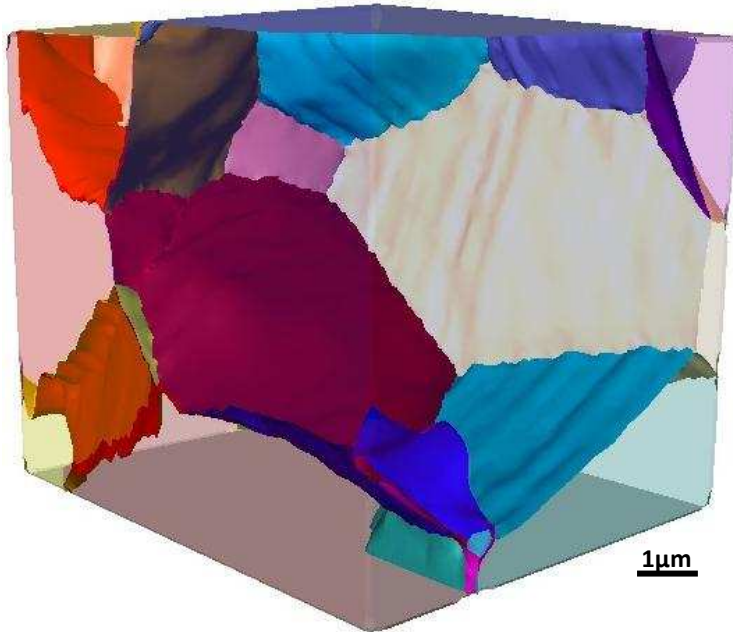


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ZrO₂ results



grain boundary reconstructions
(colors are randomly chosen)

Pictures present 3D reconstructions of grain boundaries within ceramic samples. Colors are randomly chosen.



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Presentations

- posters:
 - „3D-EBSD investigation of Ni-Mo protective coatings”
EMAS 2011, Angers
 - „Three-dimensional OIM-FIB technique: selected
application examples” 14 ICEM, Wisła
 - „Investigation of oxide layers formed on Crofer 22APU
steel” EMAS 2012, Padua



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Further research

- Further trials to obtain 3D-EBSD data from oxide layers on Crofer 22APU.
- Investigation of Crofer 22APU oxides with respect of chemical composition.
- 3D experiments with ZrO_2 ceramics with different grain sizes and its composites.
- X-ray diffraction experiments on ZrO_2 .

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