Investigation of porous microstructure of plasma-sprayed coatings

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Use: Protection of surfaces, thermal barrier

Quantification and visualization of coating microstructure:

a) better insight in the microstructure: semi-globular pores, interlamellar flat pores

b) distinction between coatings sprayed by different techniques and with variety of process parameters

c) relation between the geometry of pores and physical properties of coatings

Two parts of the talk:

(i) use of stochastic geometry for the quantification

(ii) visualization results
Condition for visualization: segmentation of microscopic images
3D information from serial sections
light or scanning electron microscopy
image analysis

Other approaches: x-ray computerized microtomography (CMT), (Kulkarni et al. 2000)
x-ray scattering (USAXS) technique, (Kulkarni et al. 2005)
small angle neutron scattering (SANS), (Ilavsky et al. 1998)
Computer-aided volumetric representation

a) Volumetric reconstruction
grinding, polishing, serial sections,
image analysis, software Lucia
3D computer reconstruction

b) Volumetric description
estimation of statistical and stereological characteristics in 3D
modelling of the porous microstructure

c) Volumetric visualization
rendering of reconstructed images
perspective figures, animations, anaglyphs
software Voxblast, Amira, Voxler
production: firing of the melt on a substrate

microstructure with spherical pores (bubbles)

estimation of the bubble density and size distribution

classical stereological approach: a single section
Wicksell corpuscule problem, Saltykov histogram method

Volenik et al (1990), Silikaty - Ceramics.
(i) plasma-sprayed coating based on ceramics

Titanates $MgTiO_3$, $CaTiO_3$, mixture with ratio $Mg : Ca = 94 : 6$

sample manufactured by water-stabilized plasma spray gun

in the Institute of Plasma Physics ASCR

feedstock powder size 63–125 $\mu m$

final coating thickness 1 $mm$

principal structural units - lamellas (splats)

result from spreading and solidification of droplets
Figure: 80 serial sections obtained by light microscopy with subsequent grinding and polishing, distant 3 $\mu m$, on each section $5 \times 5$ images
Figure: A montage of $5 \times 5$ images of a section after segmentations w.r.t. pores (white)
Classification of pores

a) semi-globular pores - almost convex, result from imperfect adhesion. Detected by morphological opening, convex structural element.

b) interlamellar flat pores - along the splats, approximately perpendicular to the spraying direction; after subtraction of semi-globular pores detected by opening with linear structural element within range of suitable orientations

c) other cracks (not investigated here)
Figure: Representation of a section of the plasma-sprayed coating with semi-globular pores (white)
2116 semi-globular pores observed (edge-correction)

2D parameters measured by image analyser: section centroid, area, minimal and maximal diameter (shape)

3D parameters reconstructed: pore centroid, volume (Cavalieri principle)
Size distribution of globular pores

Figure: Histogram of estimated volumes [\mu m^3] of semi-globular pores, pores with diameters smaller than the distance between sections are not registered.
Spatial distribution of semi-globular pores

Tests of complete spatial randomness based on centroids

a) L-function (second-order characteristics)

b) nearest neighbour distribution function
   (edge correction)

95% confidence bounds

Complete randomness rejected, mild clustering
Test based on L-function

Figure: Graph of L-function is above 95% bounds for complete randomness which results in mild clustering
Test based on the nearest-neighbour distribution function

Figure: Graph of the nearest-neighbour distribution function is on small distances to the left of 95% bounds, complete spatial randomness is rejected
Interlamellar flat pores (IFP)

Figure: Sections of IFP approximated by segments
Zero thickness assumed

602 pores detected in more than one section

Triangulation enables estimation of surface area

Shape of interlamellar flat pores

Homogeneity of IFP along the spraying direction
Interlamellar flat pores

Figure: Histogram of surface areas [$\mu m^2$] of IFP
Figure: Shapes of largest individual IFP projected onto the plane perpendicular to the spraying direction
Homogeneity of IFP

Figure: Surface areas $[\mu m^2]$ of IFP along y-axis (spraying direction)
Figure: Kernel density estimator of the number of IFP along y-axis. The size (surface area) is not considered here.
Mixed characteristics of both types of pores

a) comparison of nearest-neighbour distribution function for surfaces of semi-globular pores with distribution function of distances from semi-globular pores to the nearest IFP.

b) correlation field between the size of semi-globular pores and the location of the nearest IFP on y-axis.

Results in an overall homogeneity.
Joint characteristics of IFP and semi-globular pores

Figure: Nearest neighbour distribution functions of two types.
Joint characteristics of IFP and semi-globular pores

**Figure:** Correlation plot for the size of semi-globular pores and their location w.r.t. IFP
Quantitative description of the microstructure of coatings developed in part (i) used

Material: stainless steel AISI 316L

a) Wire-arc spraying performed at Škoda Research LtD, Plzeň, TAFA 9000 (USA) spray system

b) Water stabilized plasma-sprayed system WSP 500 (Institute of Plasma Physics, ASCR)

Main structural features: Porosity, oxide phase
Plasma sprayed coating, 3D specimen investigated

Figure: Pores black, oxides grey, basic matrix white. Spraying direction y-axis, grinding direction z-axis, 35 sections obtained distant about 5µm
**Figure:** Wire-arc sprayed coating (left), plasma-sprayed coating (right), pores white
Microstructure of oxides in a specimen section

Figure: Wire-arc sprayed coating, oxides white
Quantification of pores in AS (wire-arc sprayed) and PS (plasma sprayed) coating

Maximum pore volume AS: $6602 \mu m^3$, PS: $26016 \mu m^3$

Number of pores (volume greater than $16 \mu m^3$) AS: 814, PS: 879

Volume fraction of pores AS: 0.82%, PS: 1.92%

Volume fraction of oxides AS: 23%, PS: 30%
Figure: Wire-arc sprayed coating (left), plasma-sprayed coating (right), histograms of volume of pores
Area fraction of oxides along the section planes

**Figure:** Wire-arc sprayed coating (left), plasma-sprayed coating (right)
Conclusions from comparison of two spraying techniques

General similarity in microstructure

Lamellar structures built of metallic splats separated by oxide shells
porosity with thin cracks and irregular larger pores.

Oxidation and porosity more pronounced for plasma-sprayd coating due to higher particle temperature (accelerates formation of oxides) and their lower velocity (worse droplet spreading).

Corrosion resistant stainless steel coatings worse protection in aggressive environment
Visualizaion of 3D phases

Figure: Pores and oxides in plasma-sprayed coating, using software Voxler
Visualization of 3D phases

Figure: Pores in plasma-sprayed coating, using software Voxblast

Animations, anaglyphs - software Amira
References


Announcement

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