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Measurement of relaxation-free stress profiles in aluminium by multireflection grazing incidence X-ray diffraction with different wavelengths

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The state of residual stresses is of fundamental importance to the mechanical properties of many metals. Resulting properties are function of not only surface, but also the subsurface stress state, usually beyond the penetration depth of most X-ray sources. To circumvent this limitation conventional methods of stress profiling relies on some kind of material removal such as electropolishing or acid polishing, which are known to cause stress relaxation [1], compromising the reliability of such profiles.

Multireflection grazing incidence X-ray diffraction (MGIXD) geometry is seen as an alternative for obtaining relaxation-free, nondestructive residual stress profiles [2], potentially resulting in major revisions of the understanding of subsurface stress influence in metals' mechanical properties.

We discuss here the application of the MGIXD geometry for stress profiling in aeronautical aluminium. In this study a PANalytical Empyrean diffractometer equipped with conventional (Cu) and hard (Ag) sources was used to obtain residual stress profiles of shot peened 7050 aeronautical aluminium in depths up to 200 µm. With this results, comparisons with conventional (destructive) stress profiling methods was also made possible.

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Intergrowth of new phosphorus nitride oxide high-pressure phases elucidated using synchrotron radiation

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Whereas silicates are among the most thoroughly investigated classes of compounds, much less is known about the closely related oxonitridophosphates and phosphorus oxide nitrides. Different O/N ratios and flexible bonding to N atoms enable a rich structural chemistry, further enhanced by the possibility of including H atoms. Yet, access to new compounds in the system P/O/N/(H) is difficult and often involves high pressure and high temperature. Starting from amorphous phosphorus imide nitride oxide, various SiO_2-like modifications of PON can be obtained. Yet, syntheses at 1400 °C and up to 16 GPa yielded microcrystalline samples with complex diffraction patterns. Powder diffraction patterns being inconclusive, first information was obtained by electron diffraction and suitable crystallites of new compounds could be identified. Microfocused synchrotron radiation (ID11, ESRF, Grenoble) enables unique insight into the crystal chemistry of such compounds with unprecedented accuracy. Diffraction data from intergrown micrometer-sized crystallites correspond to the superposition of diffraction patterns of $H_3P_8O_8N_9$ [1] and new phosphorus oxide nitrides.

The compound with the idealized formula $P_{74}O_{59}N_{84}$ (monoclinic, *C*2, $V = 4878$ Å³) exhibits a 3D network of $P(O,N)_4$ tetrahedra, some sharing three vertices. $P_{40}O_{31}N_{46}$ (monoclinic, *C*2, $V = 2651$ Å³) contains similar building blocks and exhibits a higher degree of disorder. Both compounds contain a motif that is similar to the unit cell content of $H_3P_8O_8N_9$, but, in contrast, does not involve an interrupted network. This motif is characterized by 8-ring layers interconnected by additional pairs of tetrahedra. In $P_{74}O_{59}N_{84}$ and $P_{40}O_{31}N_{46}$, these patterns are interconnected by additional building blocks. These build up arrays of layers perpendicular to two crystallographic directions. These intersecting layers form channels along [010], in which the $H_3P_8O_8N_9$-like motifs are embedded. The additional layers mainly contain *vierer* rings. In $P_{40}O_{31}N_{46}$, one type of these additional layers is thicker and less corrugated than in $P_{74}O_{59}N_{84}$. Taken as a whole, all structures are mainly built up from *dreier*, *vierer*, *sechser* and *achter* rings.

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