IN-SITU DIFFRACTION STUDIES RELATED TO THERMO-MECHANICAL PROCESSES

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Introduction

The making and shaping of metals is an important process for both manufacturing products and designing the material's mechanical properties. Whether a metal exposes high strength or ductility, withstands temperature and fatigue depends minutely on its atomic and microstructure. Work-hardening, precipitation hardening, annealing, recrystallization, multi-phase compounds are examples of well employed pathways for such design. A material may have to be shapable, formable, forgeable, drawable under manufacturing conditions, while exposing high durability, ductility, strength, temperature resistance, fatigue life during application. Therefore, eminent materials research is undertaken worldwide to ever design better materials, with the aim to reduce costs in form of the amount of material and its weight in operation, production, and lifetime.

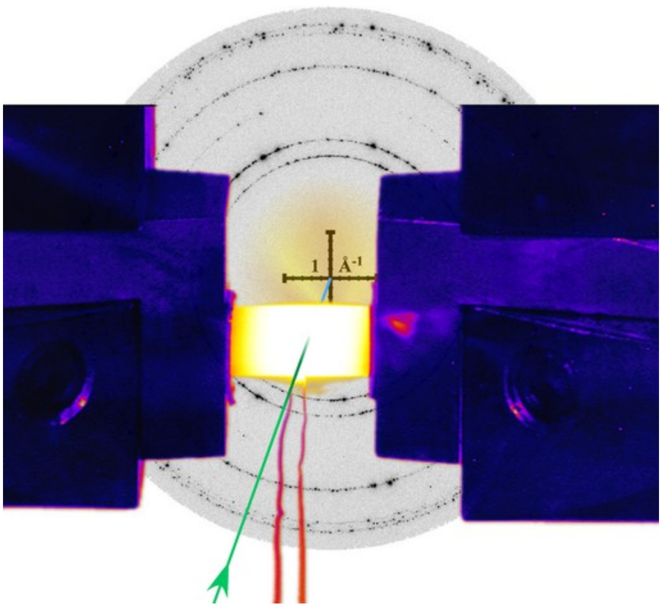

Figure 1: *The Materials Oscilloscope – in-situ diffraction measurement of a glowing sample, being compressed (foreground) while time-resolved, two-dimensional diffraction patterns are taken (background).*[1,2]

In-situ diffraction studies by penetrating neutron and synchrotron high-energy X-rays bear unique advantage to gather direct bulk information, time-resolved during heating, during plastic deformation. In a dedicated Materials Oscilloscope (Fig. 1), parameters can be adjusted in operandi to tune to the desired microstructure.[1,2]

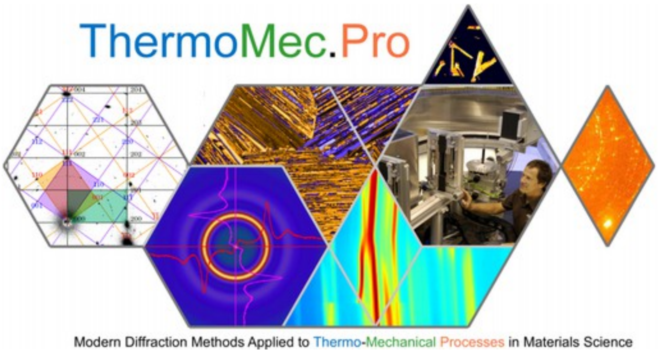

Figure 2: *The* ThermoMec.Pro *project melds the disciplines between instrumentation, materials science, physics and engineering.*[3]

Such in-situ studies bear advantage over conventional materials development, which usually occurs through a processing-and-quenching-characterization loop: A material is processed in a model or physical thermo mechanical simulation, at high temperature and given plastic deformation parameters, then quenched by dropping it into water or oil. Apart that this process is eminently slow, only near-surface volumes are probed and much of the information is changed during quench, including stress states, crystal defects, and phases undergoing transformation.

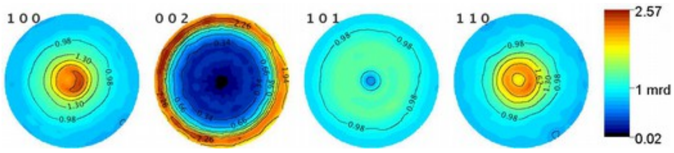

Figure 3: *Texture study of magnesium showing the basal plane normals oriented perpendicular to the extrusion axis.*[4,5]

In the current research project *Modern Diffraction Methods for the Investigation of Thermo-Mechanical Processes*, ThermoMec.Pro

(Fig 2), novel and unconventional methods, based on neutron scattering and synchrotron high-energy X-ray diffraction are being exploited in order to obtain complementary information from what is analyzed traditionally.[3] In the past two decades, neutron and synchrotron sources have developed tremendously, bearing a potential for applications and expertise yet to be exhausted. Keywords are advanced and different views of data analysis, multi-dimensional recording, time resolution and to bridge the scales from the atomic, the nano-structure, microscopic to engineering scale. It involves to meld the disciplines between expertise in instrumentation, materials science, physics, engineering and application.

Both kinds of radiation can penetrate deep into the bulk of most materials, including metals. Neutrons are well suited to gather grain-averaged information, such as phase composition, crystallographic texture (Fig. 3), macro-strain, system kinetics, lattice dynamics, while a fine and brilliant synchrotron beam can reveal information from a set of individual grains, such as their correlation, size, orientation, etc, leading to the determination of grain growth and refinement, grain rotation, activated slip systems, twinning, martensitic transformation, dynamic recovery and recrystallization.

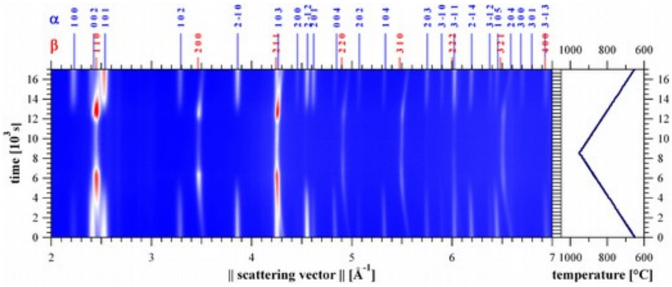

Figure 4: *Study of defect kinetics by the decrease of intensity due to extinction, described by the dynamical theory of diffraction: Time evolution of diffractograms from Zr-2.5Nb undergoing a heating-cooling cycle.*[6]

In ThermoMec.Pro, we have developed the formalism within the dynamical theory of diffraction, to interpret intensity decrease by extinction due to crystal perfection (Fig 4), which occurs upon annealing by crystal recovery, such as the annihilation of dislocations. Moreover, the crystallites are distorted again by nucleation and growth of a precipitating phase. This method allows to study the defect kinetics of metals at very high temperature.[6]

In-situ physical thermo-mechanical simulation of a titanium aluminide based alloy in a synchrotron Materials Oscilloscope revealed the distinct plastic deformation behavior of each phase in an $\alpha+\beta$-phase-field at 1573 K. While α-phase deforms by crystallographic slip and discontinued recrystallization, β dynamically recovers rapidly, constituting the 'grease' of the system.[7] Room-temperature compression of copper delivered the pathways of grain reorientation and breakage to result into a typical deformation texture.[8] Compression of magnesium at various temperatures shows the subsequent activation of twinning and different slip systems.[5] Other materials, such as twinning-induced and transformation-induced plasticity steels, zirconium alloys, and metallic glasses have been studied in a similar way.[9,10,11,12,13]

In conclusion, there is much potential in depicting neutron and synchrotron X-ray diffraction data for the understanding of materials under complex conditions, time resolved and in-situ. A dedicated Materials Oscilloscope beamline would speed up alloy and materials development by 100-1000 times.

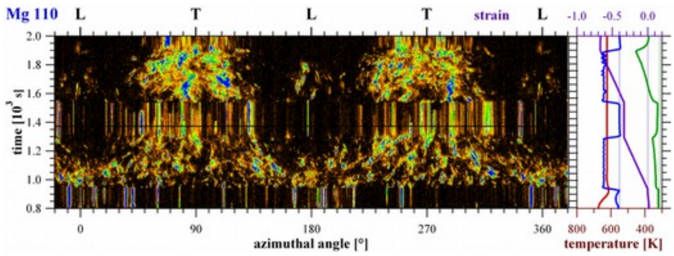

Figure 5: *Materials-Oscilloscope plot of magnesium undergoing high-temperature plastic deformation.*[5]

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