INDUSTRIAL DEVLOPMENT PROJECTS

Xray- and Neutron scattering to reveal nanoparticle evolution during critical process steps and their impact on material properties

THE INDUSTRIAL CHALLENGE

Nanoparticles and nano-precipitates strongly influence key alloy properties such as strength, corrosion resistance, electrical conductivity and magnetic behaviour. For producers of duplex stainless steels, electrical steels and aluminium alloys, controlling these features during thermal processing is essential, as even small changes in size, type or volume fraction can affect performance. With increasing use of scrap-based and circular production routes, understanding how processing steps drive nanoparticle formation or dissolution has become increasingly important.

WHY USING A LARGE-SCALE FACILITY

To capture the kinetics of nano-precipitation, how particles nucleate, grow, and transform, measurements must be performed in-situ during heating or cooling, with time resolution on the order of seconds. Such capability cannot be achieved with conventional laboratory methods. Synchrotron-based Small- and Wide-Angle X-ray Scattering (SAXS, WAXS) provide unique non-destructive access to structural changes deep inside the material, allowing us to follow precipitation processes in real time. Laboratory techniques such as TEM or APT remain crucial complementary tools, providing direct imaging and compositional insights that validate the SAXS/WAXS interpretation.

HOW THE WORK WAS DONE

The project combined ex-situ and in-situ scattering experiments at PETRA III, Hamburg. Ex-situ SAXS was performed at the P21.2 beamline, while in-situ SAXS/WAXS experiments were carried out at P07 using a modified dilatometer enabling controlled heating and cooling cycles. Samples from the industrial partners - duplex stainless steel, electrical steel, and 1xxx-series aluminum - were prepared with geometries optimized for X-ray absorption and measurement stability. In the in-situ setup, data were collected with a time resolution of 0.15 s, allowing the project to follow rapid microstructural evolution across industrially relevant thermal cycles. For selected samples where the nanoparticle volume fraction was too low to be detected using X-rays, also complementary Small-Angle Neutron Scattering (SANS) measurements were performed at the SANS-I beamline of Swiss Spallation Neutron Source, SINQ, to provide the necessary contrast and enable phase identification. These experiments were complemented by extensive laboratory analyses (SEM, TEM, and ongoing APT investigations) as well as thermodynamic simulations.

THE RESULTS AND EXPECTED IMPACT

The combined scattering techniques allowed us to track secondary phase evolution across all three alloy systems and to construct time-temperature (TTT) and continuous cooling (CCT) transformation diagrams within hours instead of weeks. This revealed formation of intermetallic and nanoprecipitate phases during thermal treatment and clarified early-stage precipitation and its link to recrystallization in aluminium. Through the SANS feasibility tests was also shown that very low-volume-fraction precipitates in electrical steels can be detected using neutrons. The results provide a solid basis for optimizing alloy chemistry, heat-treatment schedules and cooling strategies, while also clarifying which characterization methods are best suited for each material system, supporting more efficient future R&D.

The dilatometer used for in-situ SAXS/WAXS experiments at P07

"These types of measurements give data that otherwise is very time consuming if not impossible to fully document. They are also very important for understanding and optimising solution annealing parameters including cooling cycles in different production sites."
Jan Y. Jonson, Outokumpu

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