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NANO—S—MART®

Selection of wave 1 samples to perform nano segregation/precipitation and nanomechanics analysis

Deliverable 6.1

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EXECUTIVE SUMMARY

The present document constitutes Deliverable D6.1: “Selection of Wave-1 samples to perform nano- segregation/precipitation and nano-mechanics analysis”, in the framework of the Project titled “Unlocking the Effect of Residual Elements via the Nanoengineering of Sustainable Martensitic Steels” (Project Acronym: NANO-S-MART; Grant Agreement No. 1011784431).

Deliverable 6.1 documents the selection of Wave-1 material samples and associated impacted microstructural features (IMFs) for advanced nano-segregation/precipitation and nano-mechanics investigations, in line with the objectives of Task 6.1. Given the resource-intensive nature of Atom Probe Tomography (APT) and nano-mechanics testing, a phased and targeted selection approach was applied, maximising the use of knowledge generated in prior work packages.

The selected Wave 1 samples represent 24 sample-IMF combinations for APT and 6 combinations for nano-mechanics testing, identified based on the outcomes of mechanical testing, fracture analysis, advanced microscopy, and thermomechanical characterisation, complemented by insights from modelling and previous nano-scale studies. This selection ensures that the chosen samples capture material conditions before and after property deterioration, providing a robust and well-justified experimental basis for quantifying critical segregation levels and their nano-mechanical impact in the subsequent tasks.

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ABBREVIATIONS AND ACRONYMS

Abbreviations / Acronyms	Description
APT	Atom probe tomography
DQ	Direct Quenched
RAQ	Re-austenized and Quenched
EBSD	Electron-back scattered diffraction
PAGs	Prior austenite grains
IMFs	Impacted microstructural features
PAGBs	Prior austenite grain boundaries

1 Introduction

Deliverable D6.1 provides a structured and justified selection of Wave-1 material samples and impacted microstructural features (IMFs) to be investigated within Work Package 6 using advanced nano-scale characterisation and nano-mechanics techniques. Given the resource intensive nature of the techniques applied in WP6, this deliverable ensures that the nano-scale investigations focus on scientifically and industrially critical cases.

As defined in the project proposal, the Wave 1 selection within Task 6.1 comprises 24 sample-impacted microstructural feature (IMF) combinations for Atom Probe Tomography (APT) and 6 sample-IMF combinations for nano-mechanics testing. The objective is to define a targeted and representative selection of samples and impacted microstructural features (IMFs) for nano-scale investigations, acknowledging that the number of feasible measurements is limited due to the complexity and cost of techniques such as Atom Probe Tomography (APT) and nano-mechanics testing.

The selection presented in this deliverable is based on the experimental evidence generated in WP3 (Hot rolling processability), WP7 (segregation modelling) and WP4 (macro-, micro- and nano-mechanical performance), ensuring full traceability between mechanical behaviour and the nano-scale analyses planned in Tasks 6.2 and 6.3.

2 Summary of input from WP3-Hot rolling processability

WP3 provided essential process related and microstructural input supporting the selection of Wave-1 samples and impacted microstructural features (IMFs) for the nano-scale investigations foreseen in Task 6.1. WP3 focused on evaluating material behaviour under casting and hot-rolling relevant thermal and deformation conditions, thereby enabling the identification of critical material states and deformation regimes where microstructural features play a dominant role in damage initiation and property evolution.

The information generated in WP3 was used in Task 6.1 to (1) define relevant processing windows, (2) identify microstructural regions most sensitive to degradation mechanisms, and (3) support a targeted selection of representative sample conditions for nano-segregation and nano-mechanics analyses. Detailed experimental results are reported in the corresponding confidential Deliverable D3.1 and are therefore not reproduced in this public document.

2.1 Hot ductility: casting related (Task 3.1)

Task 3.1 investigated hot ductility under conditions representative of continuous casting. These studies enabled the identification of temperature ranges and microstructural states where materials are particularly sensitive to damage, especially in relation to grain boundary controlled mechanisms and transformation affected regions.

For Task 6.1, the primary input from Task 3.1 was the identification of impacted microstructural features associated with transformation related ductility degradation, which were prioritised as relevant targets for nano-scale characterisation.

2.2 Hot ductility and hot shortness: hot rolling related (Task 3.2)

Task 3.2 addressed material behaviour under reheating, oxidation and deformation conditions representative of industrial hot rolling. The investigations provided input on the combined influence of reheating conditions, oxidation behaviour, deformation mode and chemical composition on surface integrity and damage susceptibility.

For the purposes of Task 6.1, these activities enabled the identification of near surface regions and grain boundary areas as key impacted microstructural features, particularly under conditions relevant to hot-rolling induced damage mechanisms. This information guided the selection of Wave-1 samples in states where nano-scale analyses can contribute to understanding segregation and interface controlled effects.

3 Summary of input from WP4-Ranking of residual elements and identification of IMFs

WP4 provided essential input for the selection of Wave-1 samples and impacted microstructural features (IMFs) by evaluating the mechanical response of the investigated steels under diverse processing routes and heat-treatment conditions across macro-, micro-, and nano-scales.

The information generated in WP4 was used in Task 6.1 to guide the targeted selection of sample-IMF combinations for nano-segregation, precipitation and nano-mechanics investigations. Detailed experimental procedures, quantitative results and interpretations are reported in the corresponding confidential Deliverable D4.1 and are therefore not reproduced in this public document.

3.1 Macro-Mechanical Ranking of Residual Elements (Tasks 4.1– 4.3)

In Task 4.1, 4.2 and 4.3, the hot-rolled and quenched Wave-1 materials produced in WP2 were tempered in worst case conditions and tested and characterized afterwards. Tensile at room temperature and Charpy testing at different low temperatures were performed on both as-quenched and tempered materials. For a given as-quenched or tempered condition, the amplitude of the change in material properties was ranked from negative to positive to assess the impact of residual elements and processing (DQ vs. RAQ routes) on mechanical properties. These investigations enabled the identification of material states associated with altered strength and toughness levels, as well as conditions representative of baseline and degraded behaviour.

For Task 6.1, the main input from macro-mechanical testing was the identification of processing conditions and material states showing distinct macroscopic mechanical responses to residual element additions. These conditions were then selected for investigation at smaller length scales, ensuring that the nano-scale analyses focus on microstructural states most relevant to macroscopic performance differences.

3.2 Nano-Indentation and Micro-Mechanical Testing (Tasks 4.4 and 4.5)

Nano-indentation and micro-mechanical investigations in WP4 provided complementary insight into the local mechanical response of impacted microstructural features, bridging the gap between bulk mechanical behaviour and nano-scale mechanisms. These studies highlighted the relevance of localised mechanical properties at specific microstructural sites.

The input from Tasks 4.4 and 4.5 was used in Task 6.1 to identify microstructural regions where local mechanical behaviour may differ from the macroscopic response, thereby supporting the selection of IMFs suitable for nano-mechanics testing.

3.3 Multiscale Correlative Microscopy and Atomic-Scale Analysis (Task 4.6)

Multiscale correlative microscopy carried out in WP4, combining microstructural characterisation with atomic-scale analytical techniques, provided key input on the nature and relevance of interface controlled mechanisms in the investigated materials. These studies enabled the

identification of critical internal interfaces and microstructural regions that play a dominant role in governing mechanical behaviour.

For Task 6.1, the principal input from Task 4.6 was the identification of critical IMFs, such as different classes of internal interfaces, as priority targets for site-specific nano-segregation and nano-mechanics investigations. This input ensured that the selected sample-IMF combinations are representative of the most relevant interface controlled phenomena.

4 Summary of input from WP7 - Modelling residual element behaviour

Work Package 7 provides theoretical input supporting the selection of samples and impacted microstructural features (IMFs) for Task 6.1 through ab initio calculations of alloying element location preferences. Task 7.1 delivers information on the energetic tendency of alloying elements to reside in specific lattice positions, near other solutes, or at microstructural features such as grain boundaries and dislocations.

For Task 6.1, this modelling input is used to support and rationalise the experimental selection by identifying microstructural regions where segregation or clustering is thermodynamically favoured. This information complements the experimental evidence from WP3 and WP4 and helps prioritise impacted microstructural features most likely to control mechanical behaviour.

5 Wave-1 sample and IMF selection

Based on the combined qualitative inputs from WP3 (hot rolling processability), WP4 (macro-, micro- and nano-mechanical behaviour), and WP7 (ab-initio alloying element location preference modelling), a targeted selection of Wave-1 sample-impacted microstructural feature (IMF) combinations was defined. The selection focuses on microstructural regions expected to control mechanical performance, particularly those associated with interfaces, transformation-affected zones, and segregation or defect-related phenomena.

In line with the project proposal, the Wave 1 selection consists of 24 sample-IMF combinations for Atom Probe Tomography (APT) and 6 sample-IMF combinations for nano-mechanics testing.

5.1 Selected sample-IMF combinations for APT analysis (24 samples)

The following IMFs were prioritised based on WP3, WP4 and WP7 inputs:

- Prior Austenite Grain Boundaries (PAGBs)
- Packet Boundaries
- Block Boundaries
- Martensitic Lath Boundaries

Material	Processing Route	Impacted Microstructural Feature (IMF)
Ref	DQ	Prior austenite grain boundaries (PAGBs) and lath boundaries
Ref	RAQ	PAGBs and lath boundaries
Ref	DQ + T	PAGBs and lath boundaries
Ref	RAQ + T	PAGBs and lath boundaries
Cu	DQ	PAGBs
Cu	RAQ	PAGBs and lath boundaries
Cu	DQ + T	PAGBs and lath boundaries
Cu	RAQ + T	PAGBs and lath boundaries
Sn	DQ	PAGBs and lath boundaries
Sn	RAQ	PAGBs and lath boundaries
Sn	DQ + T	PAGBs and lath boundaries
Sn	RAQ + T	PAGBs and lath boundaries
Sb	DQ	PAGBs and lath boundaries
Sb	RAQ	PAGBs and lath boundaries
Sb	DQ + T	PAGBs and lath boundaries
Sb	RAQ + T	PAGBs and lath boundaries
As	DQ	PAGBs and lath boundaries
As	RAQ	PAGBs and lath boundaries
As	DQ + T	PAGBs and lath boundaries
As	RAQ + T	PAGBs and lath boundaries
P	DQ	PAGBs and lath boundaries

P	RAQ	PAGBs and lath boundaries
P	DQ + T	PAGBs and lath boundaries
P	RAQ + T	PAGBs and lath boundaries

5.2 Selected sample-IMF combinations for Nano-mechanics testing (6 samples)

Material	Processing Route	Impacted Microstructural Feature (IMF)
Ref	DQ	PAGBs
Ref	DQ+T	PAGBs
Ref	DQ+T	Martensite lath / block boundaries
Sn	DQ	PAGBs
Sn	DQ+T	PAGBs
Sn	DQ+T	Martensite lath / block boundaries