

Novel ultrahigh strength crack free tool steel by laser powder bed fusion: revisiting carbon bearing Ni–Co–Cr–Mo system

Lorena Emanuelli^{a,*}, Ville-Pekka Matilainen^b, Francesca Russo^a, Lorena Maines^a, Jonathan Hann^c, Arne Röttger^c, Massimo Pellizzari^a, Faraz Deirmina^{d,**}

^a Department of Industrial Engineering, University of Trento, Italy

^b Seco Tools, Fagersta, Sweden

^c Wuppertal University, Wuppertal, Germany

^d Sandvik Additive Manufacturing, Sandviken, Sweden

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ABSTRACT

Carbide-strengthened Ni–Co–Cr–Mo steels were developed to combine the high strength of Fe–C martensite with the weldability of Fe–Ni maraging alloys. Designed for laser powder bed fusion, Osprey® MAR55 achieves a balanced combination of printability, toughness, and strength through refined lath martensite and controlled carbide precipitation, addressing the typical weldability limitations of medium-carbon tool steels. After direct ageing at 480°C, MAR55 shows excellent properties, with toughness above 30 J, hardness around 550 HV10, and tensile strength close to 1900 MPa. Thanks to its leaner alloy chemistry, particularly reduced Ni content, and solute depletion (C, Mo, Cr) during carbide precipitation, aged MAR55 exhibits ~20% higher thermal conductivity than 18Ni300, reaching or even surpassing tempered H13 steels at elevated temperatures. In addition, carbon addition and the formation of stable Mo₂C carbides significantly enhance wear and abrasion resistance. In-situ synchrotron measurements confirm high thermal stability and slower austenite reversion compared to 18Ni300, consistent with the lower Ni content. Microstructural analysis revealed ~15 vol% retained austenite (RA) in the as-built state. Continuous-heating dilatometry and Differential Scanning Calorimetry (DSC) further revealed six transformation mechanisms during heating, involving carbide clustering, RA decomposition, alloyed cementite and carbides precipitation, Ni-driven austenite reversion, and high-temperature shear transformation. Direct tempering at 480°C reduces RA to ~3 vol% through decomposition of Mo- and C-rich austenite, while temperatures above 600°C promote Ni-driven reversion. The combination of high strength, toughness, wear resistance, thermal conductivity, and thermal stability makes MAR55 a promising material for advanced additive manufacturing and demanding engineering applications.

1. Introduction

Laser-based Powder Bed Fusion (L-PBF) has emerged as one of the most relevant technologies in metal Additive Manufacturing (AM), enabling the fabrication of three-dimensional components through the selective melting of successive thin layers of metallic powder by means of a high-energy laser beam, following a Computer-Aided Design (CAD) model [1]. One of the main advantages of this technology lies in its capability to manufacture highly complex geometries, including thin-walled structures and conformal cooling channels, which are often impossible or economically impractical to produce using conventional

manufacturing processes such as casting or forging. In addition, L-PBF offers significant benefits in terms of material efficiency due to its near-net-shape production approach, minimizing waste and allowing the reuse of unfused powder.

Despite these advantages, the industrial implementation of L-PBF remains challenging. The process generally exhibits relatively low production rates and involves extremely rapid thermal cycles, with cooling rates reaching 10⁶–10⁸°C/s. Such extreme processing conditions promote the development of high residual stresses, metastable non-equilibrium microstructures, and process-induced defects, all of which can significantly affect the mechanical performance of the final

* Corresponding author.

** Corresponding author.

E-mail addresses: lori.ema88@gmail.com (L. Emanuelli), farazdeirmina@gmail.com (F. Deirmina).

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component. Consequently, the successful application of L-PBF strongly depends not only on process parameter optimization, but also on the development and selection of alloys specifically suited to withstand these demanding processing conditions. Among the materials currently investigated for this purpose, ultrahigh-strength steels represent one of the most promising classes for tooling and high-performance engineering applications. In particular, two main classes of ultrahigh-strength tool steels have been extensively explored in L-PBF. These classes include carbon-bearing martensitic tool steels, and carbon-free martensitic maraging systems [2–4]. Carbon containing martensitic steels in AM can be classified into i) low alloy steels such as 8620 (~0.20 wt% C), 4140 and 4340 (~0.40 wt% C) [5], ii) Low to medium carbon martensitic/ferritic stainless steels such as AISI 420 (~0.20 wt% C) [6], iii) medium carbon hot work tool steels alloyed with Cr, Mo and V (or W), such as AISI H13, and H11 (~0.40 wt% C) [7] and iv) higher carbon cold work and high speed steels such as AISI M2 (~0.60 wt% C) [8]. Maraging steels, which have been exploited extensively in AM are mostly Fe–Ni–Co–Mo quaternary systems with the additions of supplementary strengthening elements such as Ti, or Al. Among them 18Ni300 maraging steel is the most commercially exploited system, comprising 18% Ni, 9% Co, 4.7% Mo and 0.6% Ti [9,10]. Stainless and Co-free maraging systems such as 13–8 PH or MAR 50 have also been studied and are being commercially used [11,12]. Medium carbon tool steels, and in particular Cr, Mo, V hot work tool steels such as H13 and H11, are characterized by an extremely hard (~600–700 HV10) quenched Fe–C martensite with a refined lath martensite substructure proportional to the carbon content [13]. Tempering of this martensite above the so-called secondary hardness peak temperature (~500–550°C) produces a tempered martensite/ferrite matrix with the dispersion of fine and stable alloy carbides [7]. This microstructure, depending on over-tempering temperature, offers a combination of good thermal conductivity, high hardness (400–550 HV10), improved toughness (Charpy V-notch (CVN) impact energy of ~15–30 J), wear-, and tempering resistance [14]. This microstructure is suited for the use of the tool at low to elevated temperature applications such as polymer injection molding, hot forging, extrusion dies, and die casting dies. However, in agreement with their poor weldability, processing of medium carbon tool steels through LAM processes stays challenging [15,16]. Cold cracking due to the residual stresses build up as a result of volume-expansive martensitic transformation in extremely hard and low-toughness fresh martensite has been reported as the main processing challenge in medium-carbon tool steels. This has been partially addressed either by plate or build chamber preheating above the martensite start (Ms) temperature. Beyond process modifications, new alloys can be designed to match process characteristics. This can be achieved by using leaner chemistries to enhance the fracture toughness of the as-built martensite and reducing the martensite/bainite hardness [17], or by applying the low transformation temperature (LTT) alloy design approach. The LTT design ensures low Ms temperatures and a high volume of retained austenite in the as-built (AB) microstructure, helping to suppress cracking by accommodating transformation stresses [18–20].

On the other hand, carbon-free Fe–Ni martensite in maraging systems is characterized by an extremely tough (CVN impact energy > 100 J) and relatively soft (~350 HV10) lath martensite with a coarse lath martensite substructure in the absence of carbon [21]. In contrast to C-bearing tool steels, substitutional elements such as Ti and Al produce age-hardening in Fe–Ni martensite upon ageing at temperatures of ~400– to 600°C [22]. Age-hardening is a consequence of precipitation of nano-size intermetallic phases (Orowan strengthening) such as Ni₃Mo or Ni₃Ti which lead to the achievement of hardness levels of 580 to 750 HV10 in combination with excellent fracture toughness (~30–70 MPa^{1/2}) at the peak ageing conditions [22,23]. Indeed, the term “maraging” was intended to convey that the precipitation reactions, responsible for high hardness of these steels, occur on ageing in the Fe–Ni martensite. As a result of the absence of carbon and the formation

of a soft martensite during quenching, these highly alloyed steels exhibit extremely good weldability. Therefore, after their prime in the 1960s–1980s, have been reemerging in industry with the growth of LAM technologies [24]. Moreover, their high hardenability and minimal dimensional changes upon aging are in favor of the whole process chain in near-net shape AM processing. However, conventional maraging steels also have some limitations for high-temperature applications. Their microstructure undergoes a diffusion-controlled reversion from martensite to austenite during heating, mainly driven by the high Ni content. The kinetics of this transformation increase with increasing Ni (or Ni-equivalent) content and ageing temperature [25], which can lead to reduced microstructural stability and tempering resistance at elevated temperatures. In addition, the absence of carbides and the relatively coarse Fe–Ni martensitic structure result in lower wear resistance than in C-bearing steels, even at high hardness levels [26]. Their relatively low thermal conductivity, typically in the range of 14–25 W m^{−1} K^{−1}, and limited high-cycle fatigue strength, reported to be around 30% of the UTS, can further restrict their use under severe cyclic thermal and mechanical loading [27,28]. Compared with maraging steels, conventional hot-work tool steels such as H11 and H13 generally offer better wear resistance and higher thermal conductivity, typically ranging from 22 to 40 W m^{−1} K^{−1}. They can reach hardness values of approximately 35–56 HRC and UTS values of 1200–1590 MPa, but their impact toughness remains relatively limited (5–40 J) and their poor L-PBF processability, mainly related to their high susceptibility to cracking, is a major drawback for additive manufacturing [22,29].

Maraging steels, in contrast, typically exhibit hardness values of 33–55 HRC, UTS values of 1000–2050 MPa and impact energies of 15–100 J, together with excellent L-PBF processability. However, their wear resistance is generally lower than that of hot-work tool steels, highlighting the trade-off between mechanical performance, thermal properties and processability. Alloy development for AM maraging steels has mainly focused on modifying the Co content. Co-free compositions have been investigated to reduce the use of critical raw materials [30], whereas Co additions have been used to increase peak hardness by reducing Mo solubility in the martensitic matrix and promoting finer and more abundant precipitates [3,4,31]. However, comparatively little attention has been devoted to improving wear resistance and thermal stability while retaining the excellent L-PBF processability of maraging steels [32,33].

In the 1970s, based on the concept of combining the advantages of standard carbon martensite with those of maraging, carbide strengthening Ni–Co–Cr–Mo steels were introduced [34,35]. These steels held up to 0.4 wt% C in combination with Ni (up to 10 wt%), Co (up to 8 wt%) and additions of Mo, Cr and V. The purpose was to offer a solution for the fabrication of large pressure vessels and hydrospace vehicles characterized by weldments of heavy sections [36]. This development stemmed from the need to combine the high yield strength and wear resistance of Fe–C martensite with good toughness, adequate weldability and hardenability of Fe–Ni martensite. Several alloys were designed to show a “dual strengthening” effect, promoted by both carbide and intermetallic precipitation. However, later investigations on commercial grades such as HY 180, AF 1410, and Aermet alloys, showed that strengthening is entirely due to carbide precipitation during tempering, and the contribution of intermetallic precipitation was only significant in the overaged condition [34,37–40]. With the emergence of laser-based AM as a viable processing route for the fabrication of tools with complex geometries and near-surface conformal cooling channels, revisiting such compositions, showing secondary hardness, which bridge the gap between high-strength Fe–C martensite and weldable Fe–Ni martensite seems to be necessary. In this context, a renewed interest has been directed towards advanced Ni–Co–Cr–Mo–C steels capable of combining the processability of maraging steels with the mechanical performance of hot-work tool steels.

Osprey® MAR55 steel (0.18 wt% C, 9 wt% Ni, 10 wt% Co, 3 wt% Cr, and 2.1 wt% Mo) has been developed to overcome the limitations of

both conventional maraging steels and medium-carbon hot-work tool steels in additive manufacturing. Its alloy design combines the excellent L-PBF processability of maraging steels, allowing crack-free fabrication without build plate preheating, with the improved wear resistance, thermal conductivity, and thermal stability associated with carbon-containing tool steels. Unlike conventional hot-work steels such as H13, which remain highly susceptible to cold cracking during L-PBF, MAR55 exhibits a tailored martensitic transformation behavior that enables defect-free processing under standard manufacturing conditions. Even if the ageing response of carbide-strengthened Ni–Co–Cr–Mo steels has been extensively investigated, a comprehensive understanding of the transformation kinetics of L-PBF processed MAR55 is still lacking. The present work combines DSC, dilatometry, activation energy analysis, in-situ synchrotron WAXS to correlate the thermal events occurring during ageing with the corresponding microstructural evolution. The interpretation of the transformation mechanisms is discussed in the context of the precipitation sequences reported for this class of alloys. Finally, the microstructural evolution is correlated with tensile properties, strength–ductility synergy, tensile toughness, thermal transport behavior, and abrasive wear resistance. Since maraging steels currently represent the reference material class for PBF-LB/M tooling applications owing to their excellent processability, the industrial-grade 18Ni300 alloy was selected as the benchmark material. The comparison highlights the benefits of introducing carbon into a maraging-type alloy design, enabling higher hardness and improved performance at elevated temperatures while preserving crack-free processability. Overall, this work provides both fundamental insight into the processing–microstructure–property relationships of L-PBF processed MAR55 and demonstrates its technological potential as a new generation of AM-ready tool steel for tooling applications.

2. Materials and methods

2.1. Powder chemical composition

The chemical composition of gas-atomized Osprey® MAR 55 tool steel (hereafter referred to as MAR55) provided by Sandvik Osprey (Sandvik, Sweden) is presented in Table 1. The particle size distribution (PSD) of the gas-atomized powder is characterized by $d_{10} = 22 \mu\text{m}$, $d_{50} = 36 \mu\text{m}$, and $d_{90} = 57 \mu\text{m}$, where d_{10} , d_{50} , and d_{90} represent the particle diameters below which 10%, 50%, and 90% of the powder volume is contained, respectively. The chemical composition was determined using high-precision analytical methods. Carbon (C) content was measured by combustion infrared absorption using a LECO analyzer (LECO Corporation, USA), while the concentrations of the other alloying elements (Ni, Co, Cr, Mo, Si, and Mn) were determined by inductively coupled plasma optical emission spectrometry (ICP-OES), in accordance with ISO/IEC 17025:2017 standards.

In Table 1, “MAR55 Nominal” refers to the target chemical composition specified by the manufacturer, whereas “MAR55 Powder” indicates the actual bulk composition of the gas-atomized powder, as experimentally measured using the aforementioned analytical techniques. For comparison, the chemical composition of 18Ni300 maraging steel is also reported in Table 1, together with its PSD values ($d_{10} = 21 \mu\text{m}$, $d_{50} = 36 \mu\text{m}$, and $d_{90} = 59 \mu\text{m}$).

2.2. Processing

In the L-PBF process, selecting appropriate process parameters is essential not only for achieving the desired mechanical properties but also for ensuring cost-effective production. To identify suitable processing conditions, an extensive experimental campaign was carried out. In total, 24 cubic samples ($15 \text{ mm} \times 15 \text{ mm} \times 15 \text{ mm}$) were produced on an EOS M290 system (EOS GmbH, Germany), equipped with a 400 W Yb-fiber laser. A comprehensive description of the L-PBF setup and manufacturing procedure is provided in the work of Chowdhury et al. [1]. All specimens were printed under an Argon atmosphere using a stripe scanning strategy with a stripe width of 5 mm. The processing parameters were intentionally varied in order to obtain different volumetric energy density (VED) conditions. The VED, expressed in J/mm^3 , is calculated according to Eq. (1).

$$VED = \frac{P}{v \cdot h \cdot t} \quad \text{Eq. 1}$$

Where P is the laser power (W), v is the scanning speed (mm/s), h is the hatch spacing (mm) and t is the layer thickness (mm). In this regard, a hatch spacing of 0.11 mm and a layer thickness of $40 \mu\text{m}$ were maintained, while only the laser power and scanning speed were varied. This parameter represents the energy input per unit volume of material and provides a normalized method for comparing different processing conditions.

The resulting final densities of the components were assessed through image analysis of their residual porosity. Fig. 1a presents micrographs obtained under varying VED conditions to achieve pore-free microstructures, while Fig. 1b illustrates the corresponding relative densities as a function of VED values.

The optimal combination of processing parameters resulted in relative densities exceeding 99.95% at a VED of 76.9 J/mm^3 . These parameters included a layer thickness of $40 \mu\text{m}$, a laser power of 340 W, a scanning speed of 1000 mm/s, and a hatch spacing of 0.11 mm. Printing was conducted without preheating the build plate or chamber and the absence of cracking was confirmed in all samples regardless of the processing parameters. The processing parameters for 18Ni300, yielding a relative density of >99.90 have been detailed in a previous study [28].

2.3. Mechanical and physical characterization

Tensile tests were carried out at room temperature using vertically fabricated cylindrical specimens, measuring 45 mm in height and 6 mm in diameter, which were machined to comply with the ASTM E8/E8M – 25 standard. Three specimens were prepared for each condition. The final tensile geometry featured a cylindrical gauge section with a diameter of 4 mm and a gauge length of 16 mm, with a fillet radius of 4 mm at the transition between the gauge section and the gripping ends to minimize stress concentration effects. Additionally, instrumented Charpy V-notch (CVN) tests were performed following ASTM E23–24 guidelines. Three specimens for each condition were initially fabricated by L-PBF with dimensions of $58 \times 12 \times 12 \text{ mm}^3$ and subsequently machined to the final standard geometry of $55 \times 10 \times 10 \text{ mm}^3$. Machining was performed to ensure full compliance with ASTM E23–24 dimensional tolerances, to achieve the required surface finish, and to remove any surface defects associated with the additive manufacturing process that could influence the impact response. The final specimens featured a 45° V-notch with a root radius of 0.25 mm, a notch depth

Table 1

Chemical composition of the MAR55 (nominal composition and that of the powder bulk) and 18Ni300 powder, wt.%.

Alloy	C	Ni	Co	Cr	Mo	Ti	Si	Mn
MAR55 Nominal	0.18	9.2	10.0	3.0	2.1	–	<0.1	<0.1
MAR55 Powder	0.18	9.00	9.90	3.20	2.30	–	0.10	0.05
18Ni300 Powder	0.02	18.00	9.10	–	4.56	0.70	0.05	0.05

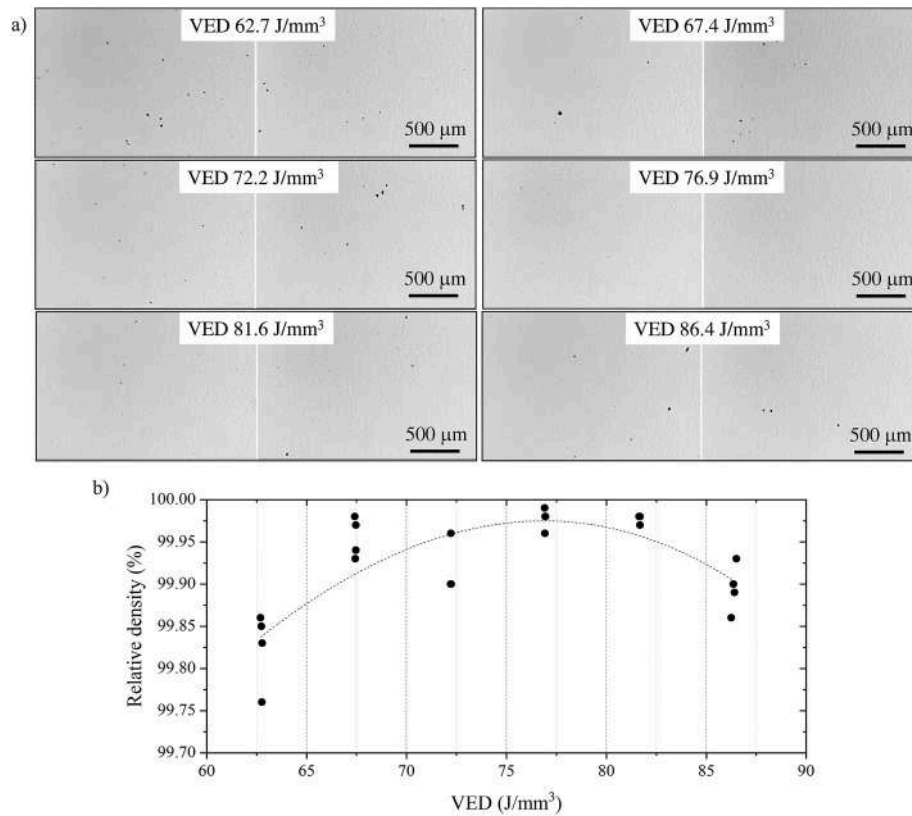


Fig. 1. a) Micrographs of cubes printed with various VED values, and b) the correlation between the achieved relative density and the applied VED.

corresponding to an 8 mm remaining ligament, and the notch positioned at the mid-length of the specimen. The notch plane was oriented perpendicular to the build direction. Both AB and aged samples were tested, and the results were compared with L-PBF processed 18Ni300 maraging steel for benchmarking purposes.

Thermal conductivity calculations were performed on AB and aged samples, at room temperature to 600°C through an indirect measurement method, according to Eq. 2.

$$\lambda = \alpha C_p \rho \quad \text{Eq. 2}$$

where thermal conductivity λ , (W/mK), can be calculated by multiplying the temperature thermal diffusivity α (m²/s) at the respective temperature, specific heat capacity C_p (J/kgK), and absolute density ρ (kg/m³). Laser Flash Analysis was performed (Netzsch LFA 467 Hyper-Flash, Netzsch –Gerätebau GmbH, Germany), to measure the thermal diffusivity. The density of the samples (7.90 g/cm³) was determined at 20°C by Archimedes' method following the standard procedure as described in ASTM B311. The temperature-dependent density up to 600°C was determined by using the dilatation records in dilatometry. For comparison purposes, the thermal conductivity results were evaluated alongside data from directly aged AM 18Ni300, direct tempered AM H13 tool steel, and quenched and tempered wrought H13, all exhibiting an ultimate tensile strength of approximately 1900–2000 MPa [17]. This comparison allows assessment of potential improvements in thermal conductivity relative to 18Ni300 and highlights how closely the values of the aged MAR55 approach or deviate from those of H13, a widely used benchmark for tooling applications.

Scratch tests were performed using an Anton Paar Step 500 MCT3 equipped with a Rockwell C diamond indenter (100 μm tip radius). L-PBF MAR55 alloys in AB and DA conditions were tested, and results were compared with L-PBF 18Ni300 in the same states to evaluate surface response under repeated contact loading. Samples were ground with 1200-grit SiC paper and polished with a 3 μm diamond suspension,

achieving an R_a of 0.5 ± 0.1 μm for all samples.

Bidirectional multipass scratch tests were conducted for 25, 50, and 100 cycles, keeping track length, sliding speed, and normal load constant at 5 mm, 200 mm/min, and 15 N. Wear tracks were examined with an Olympus DSX1000 optical microscope, and three cross-sectional profiles per condition were extracted from 3D images.

Wear volume was determined by subtracting the pile-up at the groove edges from the groove volume, since plastic deformation at the sides does not represent material loss. Wear volumes after 25, 50, and 100 cycles (sliding distances of 125 mm, 250 mm, and 500 mm) were used to analyze the wear behavior. Specifically, the initial run-in stage (0–25 cycles), corresponding to the optimization of contact conformity, and the subsequent steady-state stage (25–100 cycles), representing the main tribological regime predominantly governed by abrasive wear, were evaluated by plotting wear volume versus sliding distance. The Archard wear law was applied to calculate the abrasive wear coefficient (Kabr) for both stages [41].

2.4. Materials characterization

Dilatometric tests were performed using a Bahr DIL 805 dilatometer (TA Instruments, USA) with a heating rate of 10 °C/min under a dynamic vacuum (5×10^{-4} mbar). Cooling stage was carried out under argon flow. Isochronal heating tests were conducted up to 900°C at 4 °C/min to highlight all the different transformation occurring during heating, namely precipitation phenomena, austenite reversion due to Ni diffusion and shear transformation of residual martensite (ferrite) into austenite [3,42–44]. Different low heating rates (1 °C/min, 2 °C/min, 4 °C/min, 6 °C/min and 10 °C/min) were selected to study the precipitations and austenite transformations by measuring their activation energies. The dilatometric strain (Δ) was differentiated with respect to time ($d\Delta/dt$) to emphasize the different phase transformation contributions and identify the temperature peaks of the transformations.

DSC measurements were carried out using a Mettler Toledo DSC 5+ system (Mettler Toledo, USA) under a nitrogen atmosphere over the temperature range 25–700°C. Samples (30–80 mg), extracted from 4 mm diameter cylindrical specimens, were analyzed in heat-flux mode using heating rates of 2, 5, 10, and 15°C/min. Activation energies were determined using a Kissinger-like analysis based on the transformation peak temperatures [45,46] (Eq. (3))

$$\ln \left(\frac{T_f^2}{\phi} \right) = \frac{E}{RT_f} + \text{constant} \quad \text{Eq. 3}$$

Where R represents the gas constant (8.314 J/mol K), T_f is the transformation temperature in K corresponding to a specific fraction f of phase transformation, and ϕ is the heating rate in K/min. Typically, T_f refers to the peak temperature, and in this work, it was evaluated by means of the tangent method [47].

A combined dilatometry–DSC approach was adopted to obtain a comprehensive description of the phase transformations during heating. Dilatometry allowed the detection of dimensional changes associated with phase transitions, while DSC provided complementary thermal signals associated with these phenomena, both endothermic and exothermic.

Tempering resistance and aging behavior were assessed by measuring hardness after 1 h and 2 h at temperatures of 480°C, 525°C, 600°C, and 650°C. These heat treatments (HTs) were conducted in a laboratory muffle furnace under an air atmosphere, with temperature monitored using a thermocouple placed on a reference sample. Longer holding times, namely 4 h, 10 h, 20 h, and 40 h were considered for tempering at 480°C, 600°C and 650°C to highlight the tempering behavior and the softening effect due to dislocation recovery, precipitate coarsening, and austenite reversion. For the systematic benchmarking of mechanical and physical properties, the Direct Aged (DA) condition was defined as peak aging at 480°C for 2 h for the MAR55 alloy. For comparison, 18Ni300 samples were aged under the same condition. All specimens were air-cooled to room temperature after the isothermal hold. Vickers microhardness measurements were performed in accordance with ASTM E92-23, using an EMCO-TEST M4U 025 hardness tester (EMCO-TEST Prüfmaschinen GmbH, Austria). A load of 10 kgf and a dwell time of 10 s were applied.

Microstructural characterization was conducted following appropriate metallographic preparation. Nital 2% etching, consisting of ethanol 96% and 2 vol% of nitric acid 65% was used to evaluate the general microstructure [48]. Lichtenegger and Bloch's colored etchant (20 g ammonium bifluoride, 0.5 g potassium disulfite, and 100 mL warm distilled water) was used to highlight the chemical inhomogeneity and the evolution of solidification morphology by comparing as-built and 600°C tempered samples at different isothermal holding times [48]. The microstructure was examined using Light Optical Microscopy (LOM, Zeiss, Germany) and Scanning Electron Microscopy (2SEM, JEOL, Japan). A JEOL Ltd. IT300 SEM equipped with a Bruker Corporation XFlash 630 M energy-dispersive X-ray spectroscopy (EDXS) detector, featuring an active detector area of 30 mm², was used. To evaluate chemical micro-segregation in the AB and treated samples by EDXS analysis, an accelerating voltage of 15 kV and an acquisition time of 10 s were employed. To evaluate the chemical micro-segregation with EDXS in the AB and treated samples, a voltage of 15 kV and an acquisition time of 10 s were set. Phase maps of AB and DA MAR55 were obtained using Field Emission Gun Scanning Electron Microscopy (FE-SEM, Zeiss Sigma, Germany) equipped with an Electron Backscatter Diffraction (EBSD) system.

An electropolishing (EP) was performed in a stirred solution of 400 ml distilled water and 600 ml sulfuric acid (95–97%) with a constant voltage of 6 V DC for 20 min at room temperature, using a titanium cathode coated with ruthenium dioxide, before the X-ray diffraction (XRD) analysis to remove any effects due to mechanical grinding, such as stress-induced martensite transformation, which could affect the final

ex-situ XRD evaluation. The ex-situ XRD measurements were conducted using a Rigaku D/Max-B diffractometer (Rigaku Corporation, Japan) using Cu K α radiation (Cu K α_{avg} = 1.54184 Å). The setup included Bragg–Brentano para-focusing geometry, a diffracted beam monochromator, and a standard copper target X-ray tube operating at 40 kV and 30 mA. The data were collected over a 30° to 100° diffraction angle (2 θ).

To examine the microstructural evolution of MAR55 and 18Ni300, high-resolution in-situ high-temperature WAXS measurements were conducted at the DELTA synchrotron facility, Beamline 9, at the Technical University of Dortmund, Germany. Samples were carefully prepared to minimize mechanical deformation, first by grinding and then polishing to a final surface finish of 1 μm . The experiments were performed under an argon atmosphere using a DHS 1100 graphite dome in combination with a TCU-200 temperature controller (Anton Paar, Austria). Temperature increments were applied in discrete steps, ranging from room temperature up to 650°C. At each target temperature, samples were held isothermally for 6 min prior to data acquisition. The total measurement time for each step was 12 min. The X-ray beam had an energy of 20.0 keV (wavelength = 0.619921 Å) and was directed at the sample surface with an incidence angle of 3°, featuring a beam footprint of 0.1 $\times$ 0.8 mm² (width $\times$ height). Diffraction patterns were collected using a MAR345 image plate detector (marXperts, Germany) [49]. Before the experiments, calibration was performed at room temperature using a CeO₂ standard under identical conditions to determine instrument-specific parameters such as the detector-to-sample distance and the beam center. Two-dimensional Debye-Scherrer rings recorded during the experiments were integrated into one-dimensional intensity versus 2 θ plots using Fit2D software and appropriate integration masks [50]. All the diffraction spectra were analyzed using a combined Rietveld method via MAUD software (Materials Analysis Using Diffraction, Italy) [51]. The initial crystallographic data for ferrite and austenite were retrieved from the MAUD phase database and verified against the Crystallography Open Database (COD).

2.5. Thermo-Calc simulation

The equilibrium step diagrams for MAR55 and 18Ni300 maraging steel were generated using Thermo-Calc (Thermo-Calc Software, Sweden) with the TCFE12 database. The simulations were performed with a molar quantity of 1 and a pressure of 10⁶ Pa. The equilibrium step diagrams provided in Supplementary File 1 were used to calculate the equilibrium austenite content at approximately 600°C for both MAR55 and 18Ni300 (23 vol% and 50 vol%, respectively). These values were then compared to the measured austenite content obtained after tempering MAR55 at 600°C for 40 h, as well as to existing literature data for 18Ni300 [44]. This comparison helped assess how the austenite content in MAR55 after tempering correlated with the data available for 18Ni300, providing a reference for understanding the behavior and transformation of these materials under similar conditions. Additionally, the partitioning of elements in austenite as a function of temperature was calculated for both steels, with the corresponding graphs provided in Supplementary File 1. Thermo-Calc, combined with the Diffusion Module (DICTRA) and the MOBFE7 database, was utilized to investigate and predict micro-segregation (solute redistribution) during solidification in L-PBF at the intercellular regions. This analysis included evaluating variations in martensite start (Ms), 50% Martensite formed (M50) and 90% Martensite formed (M90) temperatures, from the core of the cell to the intercellular zone by using property module in Thermo-Calc.

3. Results

3.1. As-built microstructure

Micrographs of the AB MAR55 sample etched with Nital 2% are

shown in Fig. 2.

From the LOM micrograph (Fig. 2a), characteristic laser tracks, specifically melting pool boundaries (MPBs), are visible. The SEM micrograph (Fig. 2b) highlights the typical cellular solidification morphology of the L-PBF processed alloys. Chemical inhomogeneity is evident in the SEM micrograph in Fig. 2c. Specifically, using back-scattered electrons (BSE), the intercellular areas (2) appear brighter, suggesting a higher concentration of heavier elements. However, the semi-quantitative EDXS analysis (Table 2) does not show a marked difference in composition. This apparent discrepancy can be explained by the limited spatial resolution of EDXS: due to the very small size of the intercellular regions, even at reduced accelerating voltage, the electron interaction volume likely extends beyond the targeted area, incorporating signal from the surrounding matrix. As a result, the measured composition represents an average of adjacent regions, potentially masking local compositional differences.

Nevertheless, a slightly higher concentration of Mo is detected in the intercellular regions, accounting for the brighter appearance of these areas in the BSE micrograph in Fig. 2c. The intercellular micro-segregation may lead to the stabilization of retained austenite by a decrease in the martensite start temperature.

This effect is further supported by Scheil solidification modeling with the DICTRA module, using MAR55 nominal composition (Table 1), and a solidification cell size of $\sim 1 \mu\text{m}$ (Fig. 2c). By plotting the concentration profiles of alloying elements as a function of the distance from the cell core to the intercellular micro-segregation in parent austenite (Fig. 3a), it becomes evident that the concentration gradients of C and Mo are steeper compared to Cr, Co, and Ni. A slight decrease in the concentration of Mo and Cr in the austenite very close to $0.5 \mu\text{m}$ (i.e., at the final liquid-to-solid transition) can be attributed to the precipitation of Mo- and Cr-rich carbides, as predicted by Scheil solidification modeling. Furthermore, by plotting the martensite start temperatures (Ms), and the M50 and M90 temperatures as a function of these concentration gradients using the property module in Thermo-Calc, it is predicted that approximately 8% RA will be stabilized in the region of micro-segregation (i.e., starting at around $0.45 \mu\text{m}$), where the Ms, M50, and M90 temperatures significantly decrease due to C and Mo enrichment (Fig. 3a). To confirm the presence of RA experimentally XRD was performed. Electropolishing was performed before the analysis to remove any effects due to mechanical grinding, such as stress-induced martensite transformation, which could affect the final XRD evaluation. XRD spectra before and after EP of the AB sample are shown in Fig. 3b. The presence of RA noticeably increases after electropolishing, as evidenced in Fig. 3b. The unaffected RA content in the as-built condition, measured by XRD analysis, is approximately 15 vol%. This reveals the occurrence of stress-induced martensitic transformation during mechanical grinding and polishing. Electropolishing effectively removes the altered surface layer, resulting in an increase in retained austenite content from 3 vol% to 15 vol%. The use of Cu K α radiation in XRD

Table 2

EDXS analysis in wt.% of spots 1 and 2 shown in Fig. 1c, acquired with 15 kV and 10 s of acquisition time to highlight the chemical discrepancies.

Element/Position	Fe	Ni	Co	Mo	Cr	Si
Position 1	Bal.	7.8	10.2	2.1	3.8	0.1
Position 2	Bal.	7.8	10.5	2.6	4.0	0.1

analysis, with its limited penetration depth of approximately $6 \mu\text{m}$ in iron-containing samples (at a typical Bragg angle of $2\theta \approx 35^\circ$) [52], emphasizes the necessity of removing the altered surface layer to ensure accurate phase quantification and residual stress measurements. The presence of intercellular RA, of around 10 vol%, was also confirmed through EBSD phase analysis (Fig. 3c) [53]. Inverse pole figure maps along the Y-direction (IPF-Y) clearly demonstrate that the crystallographic orientation of RA is related to that of the parent prior austenite grains (PAGs). Meanwhile, IPF-X maps reveal the hierarchical structure of lath martensite, including lath, block, and packet boundaries, within the PAGs, along with serrated block boundaries. The model predictions are in good agreement with the experimental results regarding the position and composition of RA. However, discrepancy is observed in terms of RA volume fraction ($\sim 8\%$ by modeling vs. $\sim 10\%$ by EBSD vs. $\sim 15\%$ by XRD). It is well established that, in addition to micro-segregation during solidification, the characteristic thermal cycling of the layer-by-layer deposition in L-PBF can induce further partitioning effects, leading to enhanced stabilization of RA in the final microstructure [28,54]. These effects were not taken into account in the present model. Moreover, the thermodynamic model used in Thermo-Calc cannot fully capture the complex interplay of kinetics, partitioning, and local compositional heterogeneities that occur during rapid solidification and repeated thermal cycling, as seen during L-PBF.

3.2. Hardness evolution and microstructural changes during ageing

The ageing curve was determined following a 2-h isothermal hold at different temperatures (Fig. 4a). The phase composition of MAR55 at the secondary hardness peak (DA) was then analyzed by XRD and compared with that of MAR55 in the AB condition (Fig. 4b).

The tempering curve shows in Fig. 4a highlights the presence of a secondary peak in hardness around 500°C , after which there is a drop in hardness at higher temperatures.

XRD patterns (Fig. 4b) indicate a significant reduction in RA after ageing as demonstrated by Paul A. Davies et al. [53]. Although no diffraction peaks attributable to carbide phases are visible in the XRD pattern of the DA sample, this is likely due to the intrinsic limitations of the technique. The carbides formed during direct aging are expected to be nanometric and present in a very low volume fraction. As a result, their diffraction signal is too weak and broadened, and further affected by peak overlap with the dominant matrix reflections, to be resolved using conventional XRD.

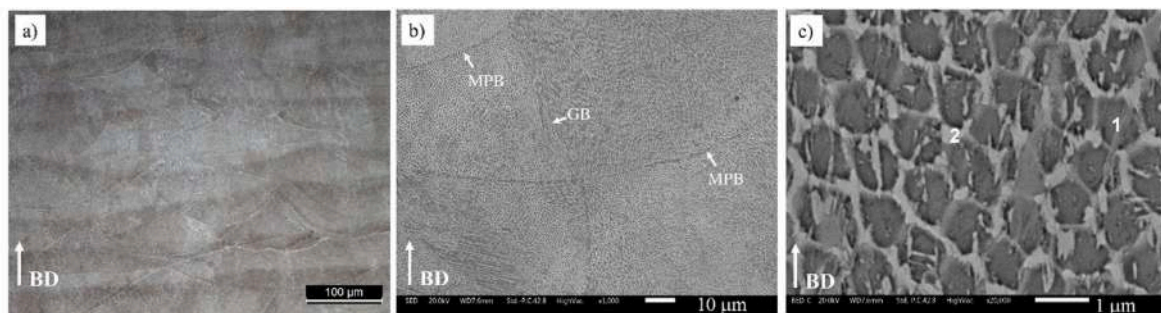


Fig. 2. AB MAR55 a) LOM micrograph highlighting melt pool boundaries, b) SEM image acquired in secondary electron mode showing melt pool boundaries (MPBs), grain boundaries (GBs), and the cellular solidification structure and c) SEM image acquired in backscattered electron mode at higher magnification, used to evaluate chemical inhomogeneity between cellular and intercellular regions.

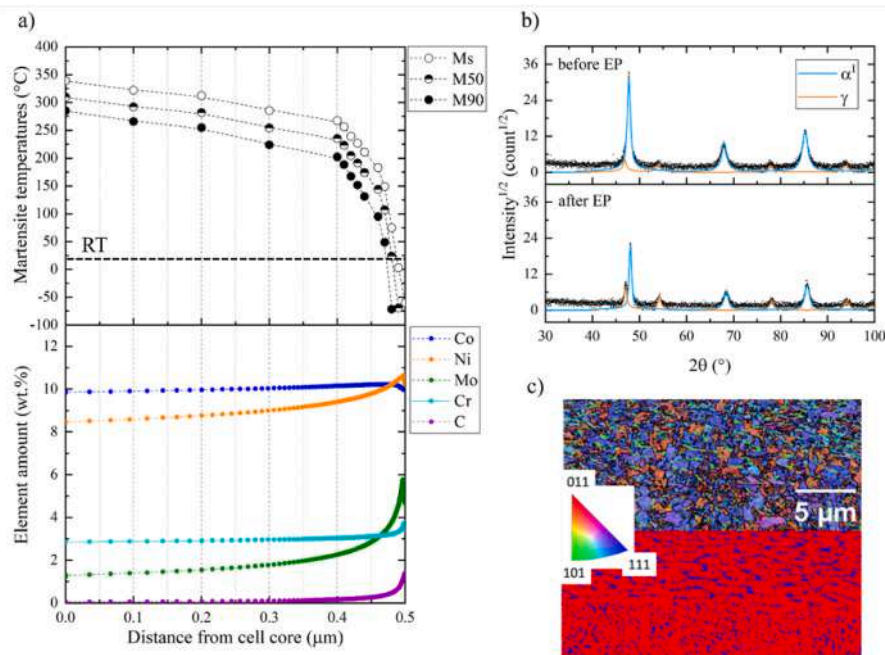


Fig. 3. (a) Thermo-Calc simulation of elemental distribution profiles and their consequent influence on martensite start temperatures from the cell core to the intercellular regions. Fe is omitted to highlight segregation of alloying elements. (b) XRD spectra acquired after electropolishing. (c) Inverse Pole Figure (IPF-X) map of AB MAR55 illustrating the crystallographic orientations of martensite laths and RA, together with a phase map identifying RA (blue) in the intercellular regions. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

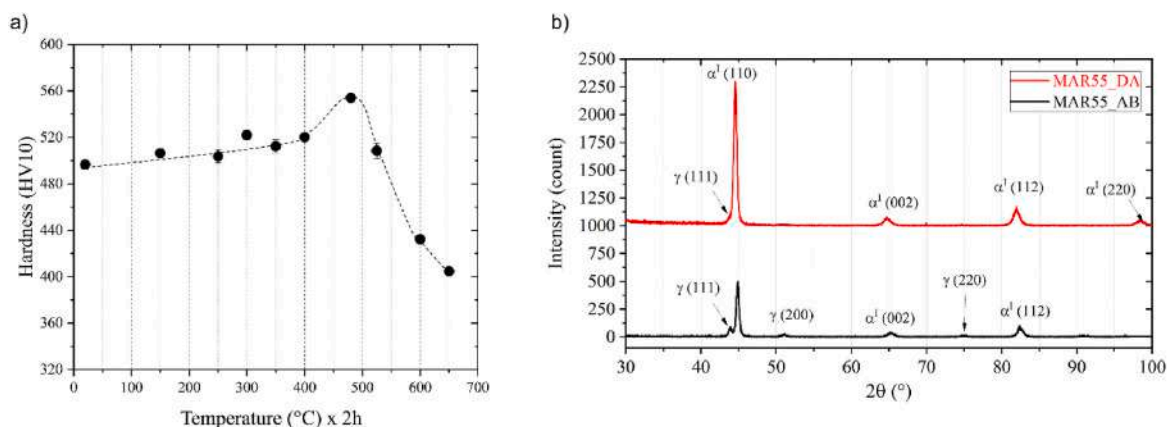


Fig. 4. a) tempering curve with a dwell time of 2 h at each tempering temperature and b) XRD spectra of MAR55 in as built state and after ageing at the secondary hardness peak (480°C).

The ageing behavior was further evaluated by hardness measurements after extended holding times (4–40 h) at 480°C, 600°C, and 650°C (Fig. 5).

The curves shown in Fig. 5 illustrate the ageing behavior during tempering at 480°C. After 1 h at this temperature, the hardness increases by approximately 60 HV10, reaching a peak value of around 554 HV10. Extending the holding time to 2 results in stable hardness values, indicating that peak hardness is achieved between 1 and 2 h at 480°C. However, further increasing the temperature to 600°C and 650°C results in a noticeable decrease in hardness. Extending the holding time at 480°C results in a decrease in hardness of approximately 20 HV10 from 2 to 4. Beyond this period, the hardness stabilizes at 535 HV10, maintaining this value for up to 40 h. At a higher ageing temperature of 600°C, a hardness reduction of approximately 88 HV10 is observed after 4 h, followed by a further decrease of 44 HV10 from 4 to 40 h, as shown in Fig. 5. During ageing at 650°C, an initial drop in hardness of 113 HV10 occurs within the first 4 h, with an additional decrease of 46 HV10

from 4 to 40 h.

3.3. Effect of direct ageing on mechanical and physical properties

To assess the effect of heat treatment on MAR55, its mechanical and thermal properties in the AB condition were evaluated and compared with those after DA at the secondary hardness peak (480°C for 2 h), as indicated by the ageing curve in Fig. 4a. For reference, tensile and CVN measurements of MAR55 in both AB and DA states were benchmarked against 18Ni300 fabricated via additive manufacturing under the same conditions [28].

Room-temperature tensile tests were performed on all samples, and the average stress-strain curves from three tensile tests per condition are presented in Fig. 6.

Additionally, CVN impact tests were conducted to assess the effect of direct ageing on the toughness of MAR55. Consistent with the tensile tests, the results were compared to those of 18Ni300 steel in both AB and

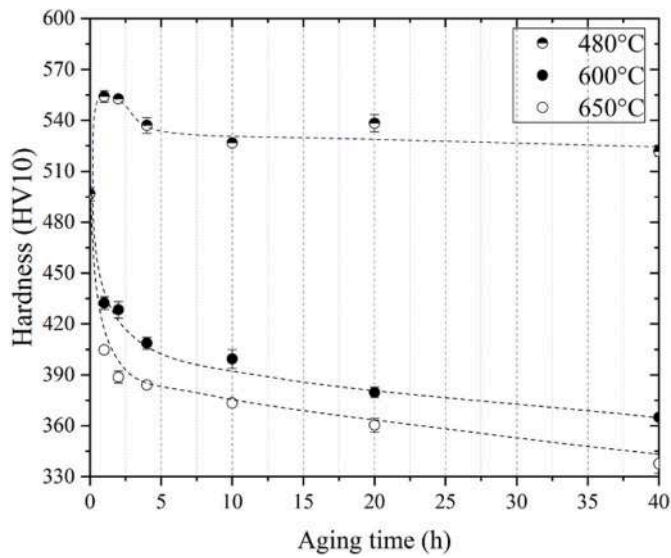


Fig. 5. Isothermal tempering resistance of MAR55 at extended holding times, specifically 4, 10, 20, and 40 h, at 480°C, 600°C, and 650°C.

DA conditions [28]. All data are summarized in Fig. 7. Specifically, Fig. 7a presents the average values and standard deviations of yield strength ($YS_{0.2}$), UTS, and maximum elongation at rupture ($\epsilon_{rupture}$) for MAR55 and 18Ni300 in both AB and DA states. In contrast, Fig. 7b shows the corresponding CVN impact energies.

In the AB condition, MAR55 exhibits a $YS_{0.2}$ of 1496 ± 41 MPa, a UTS of 1626 ± 7 MPa, and an $\epsilon_{rupture}$ of $17.9 \pm 0.3\%$. The CVN impact energy is 58 ± 1 J. In comparison, AB 18Ni300 displays a $YS_{0.2}$ of 1179 ± 19 MPa, UTS of 1189 ± 2 MPa, an elongation of $18.2 \pm 0.1\%$, and a CVN impact energy of 108 ± 3 J.

After DA, MAR55 reached a $YS_{0.2}$ 1678 ± 12 MPa, a UTS of 1898 ± 15 MPa, and an $\epsilon_{rupture}$ of $11.2 \pm 0.5\%$, and a CVN impact energy of 32 ± 3 J. For 18Ni300 in the DA condition, a $YS_{0.2}$ 1971 ± 20 MPa, a UTS of 2021 ± 6 MPa, an $\epsilon_{rupture}$ of $9.9 \pm 0.2\%$ and CVN impact toughness of 17 ± 1 J.

To further assess the mechanical performance of MAR55, the strength–ductility balance ($UTS \times \epsilon_{rupture}$) and tensile toughness ($(YS_{0.2} + UTS)/2 \times \epsilon_{rupture}$) were evaluated by means of simplify equations and summarized in Table 3.

The strength–ductility balance provides a practical metric for assessing the overall mechanical efficiency of a material by simultaneously accounting for load-bearing capability and plastic deformability prior to failure [55]. In parallel, the estimated tensile toughness provides a simplified measure of the energy absorbed during tensile deformation

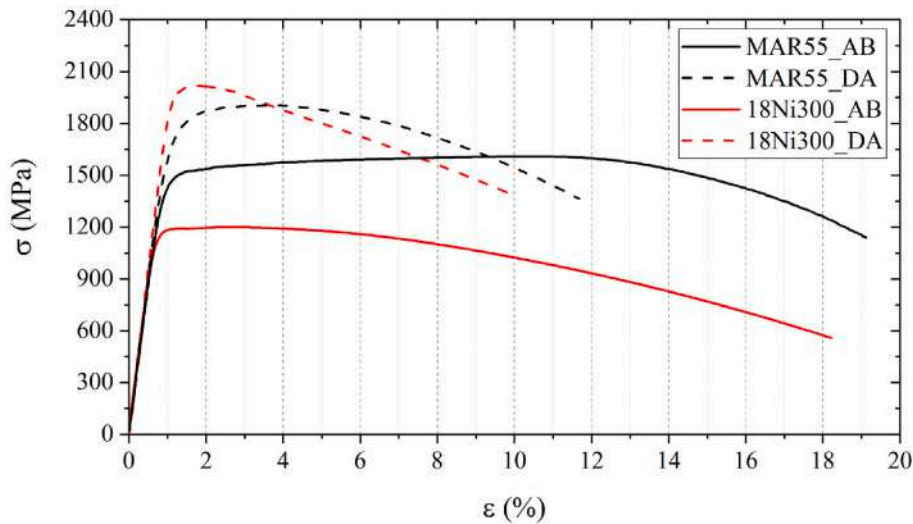


Fig. 6. Tensile curves of MAR55 in AB and aged condition compared with that of 18Ni300 [28].

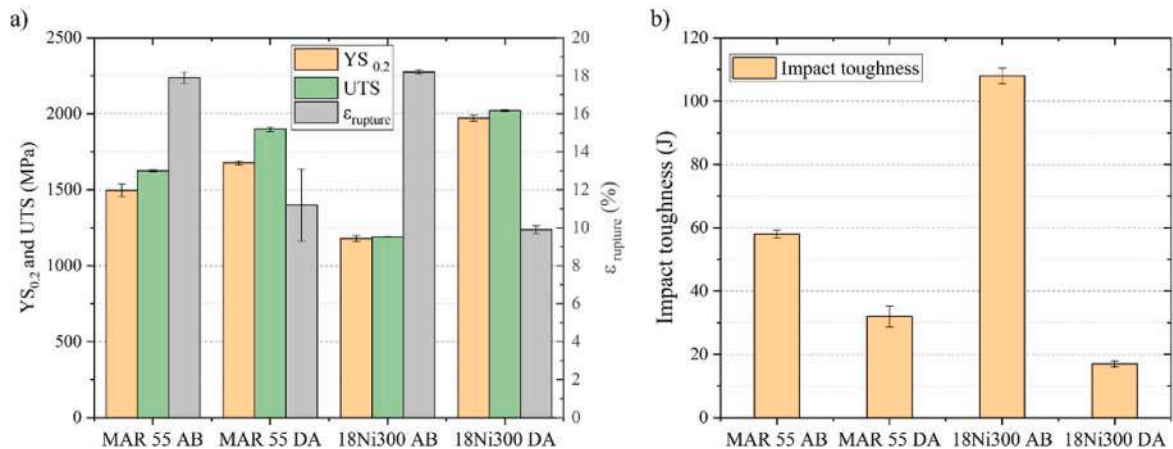


Fig. 7. a) Tensile properties and b) CVN impact energies of MAR55 in AB and aged condition compared with those of 18Ni300 [28].

Table 3

Comparison of strength–ductility balance and estimated tensile toughness values for the MAR55 benchmark 18Ni300 alloys in the AB and DA states.

Materials	strength–ductility balance (MPa%)	tensile toughness (MPa%)
MAR55 AB	29105 ± 504	27942 ± 598
MAR55 DA	21258 ± 964	20026 ± 900
18Ni300 AB	21640 ± 124	21549 ± 209
18Ni300 DA	20008 ± 408	19760 ± 411

up to fracture, approximating the area under the engineering stress–strain curve. Together, these parameters enable a more comprehensive evaluation of mechanical performance beyond the separate consideration of strength and elongation. In the AB condition, the MAR55 alloy exhibits a strength–ductility balance of 29105 ± 504 MPa%, exceeding that of the benchmark 18Ni300 AB alloy (21640 ± 124 MPa%) by $\sim 34\%$. Following the framework proposed by Li et al. [55], this improved synergy can be attributed to the combined effect of increased resistance to dislocation glide associated with the carbon-containing martensitic matrix and a more homogeneous slip distribution promoted by the refined cellular substructure and the presence of retained austenite (~ 15 vol%). Consistently, MAR55 AB shows a tensile toughness of 27942 ± 598 MPa%, significantly higher than that of 18Ni300 AB (21549 ± 209 MPa%), indicating a higher capacity for energy absorption prior to fracture. After DA, both alloys exhibit a reduction in these parameters, reflecting the shift in strengthening mechanism toward precipitation hardening. Nevertheless, MAR55 DA maintains a higher strength–ductility balance (21258 ± 964 MPa%) than 18Ni300 DA (20008 ± 408 MPa%), despite showing comparable values to 18Ni300 AB. Notably, this level of mechanical efficiency is achieved at a substantially higher strength level (UTS ~ 1900 MPa compared to ~ 1190 MPa for 18Ni300 AB), highlighting the retained synergy between strength and ductility in the MAR55 system after ageing. The tensile toughness of MAR55 DA (20026 ± 900 MPa%) remains comparable to that of 18Ni300 DA (19760 ± 411 MPa%), despite differences in absolute strength levels. Overall, these results suggest that the transition from intermetallic strengthening in 18Ni300 to carbide-based

strengthening (Mo_2C) in MAR55 promotes a more balanced combination of strength and energy absorption capability. Fig. 8 shows the fracture surfaces of MAR55 tensile specimens in both the AB and DA conditions. For the benchmark 18Ni300 alloy, fracture surface analysis is not repeated in the present study, as the reference data adopted for comparison were taken from the previous work of Deirmina et al. [28].

In the AB condition (Fig. 8a and b), MAR55 exhibits a macroscopic cup–cone fracture morphology, which is a classic indicator of high global ductility. At higher magnification, the surface is characterized by a dense population of ductile dimples (Fig. 8b), resulting from the nucleation, growth, and coalescence of micro-voids during plastic deformation. This morphology is consistent with the high elongation ($\sim 17.9\%$) and exceptional Charpy V-notch (CVN) impact energy (58 ± 1 J) recorded for the AB state. When compared to the 18Ni300 AB benchmark reported by Deirmina et al. [28], both alloys show highly ductile behavior. However, MAR55 AB achieves this while maintaining a significantly higher yield strength (~ 1496 MPa vs. ~ 1179 MPa for 18Ni300 AB). Following DA at 480°C , the fracture surface of MAR55 (Fig. 8c and d) remarkably preserves a predominantly ductile fracture character, despite the UTS increasing to approximately 1900 MPa. Compared to the AB condition, the DA fracture surface exhibits finer dimples and a less pronounced reduction in area, consistently reflecting the expected decrease in ductility after ageing. In addition, several secondary cracks can be observed, a characteristic feature frequently reported in ultra-high-strength alloys (~ 1900 MPa), where the large amount of elastic strain energy accumulated during loading is rapidly released upon fracture, leading to crack propagation along secondary fracture planes. Nevertheless, the persistence of a predominantly dimpled morphology confirms that the alloy retains a significant capacity for plastic deformation and energy absorption prior to final failure. This is quantitatively supported by the CVN toughness of 32 ± 3 J.

The thermal conductivity of MAR55 in both AB and DA conditions was measured and compared with 18Ni300 in DA states [56] as well as with H13 hot-work tool steel, direct tempered (DT) and wrought H13 in the quenched and tempered condition (QT) [57], as shown in Fig. 9. The reference data for both H13 correspond to samples exhibiting an

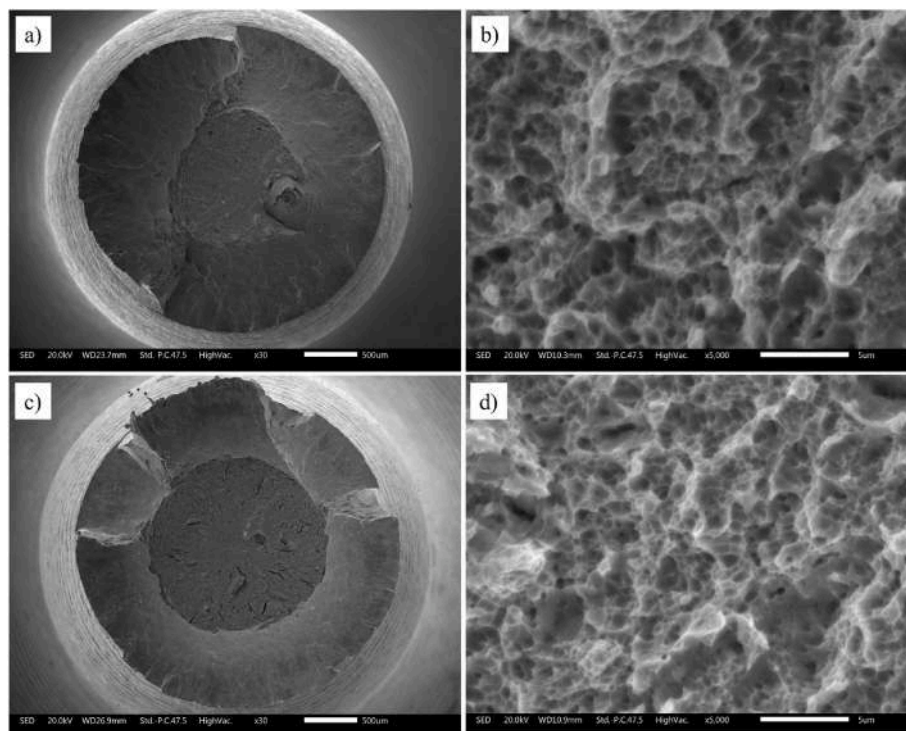


Fig. 8. SEM micrographs showing the fracture surfaces of MAR55 AB (a) at low and (b) high magnification, and of MAR55 DA (c) at low and (d) high magnification.

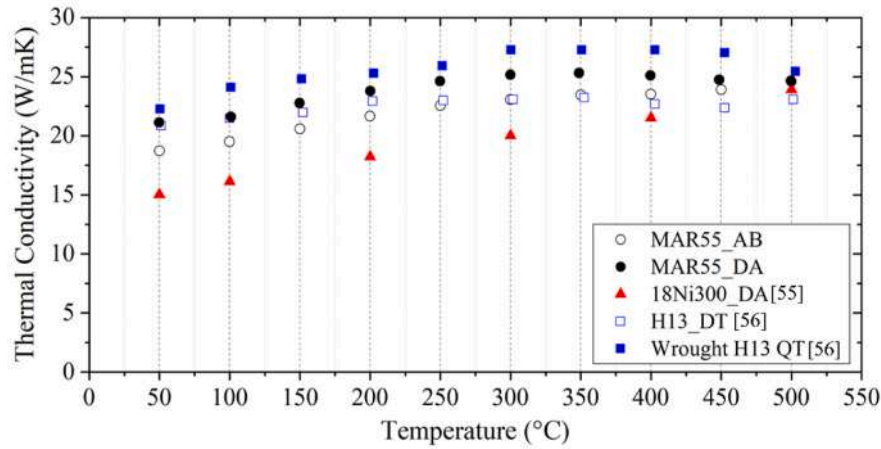


Fig. 9. Measured thermal conductivity of MAR55 in AB and DA conditions, compared with literature data for peak-aged 18Ni300 [55], H13 tempered at the peak hardness temperature produced via additive manufacturing, and wrought H13 in the quenched and tempered condition [56].

ultimate tensile strength of approximately 1900–2000 MPa, compared to MAR55, which ranges from about 1600 MPa in the AB condition up to around 1900 MPa in the DA condition at peak hardness.

The results indicate that 18Ni300 in the DA state exhibits the lowest thermal conductivity across the tested temperature range, although at 500°C it reaches values comparable to those of MAR55. The MAR55 AB alloy shows higher conductivity, which further increases after direct ageing at 480°C. As expected, the wrought H13 QT specimen exhibits

the highest thermal conductivity at room temperature. However, the additively manufactured H13 DT condition consistently shows slightly lower conductivity than the wrought counterpart. The thermal conductivity of aged MAR55 remains comparable to that of the additively manufactured tempered H13 up to approximately 100°C. At higher temperatures, MAR55 exhibits higher conductivity values, indicating an improvement in thermal transport with increasing temperature. Nevertheless, even at elevated temperatures, MAR55 maintains

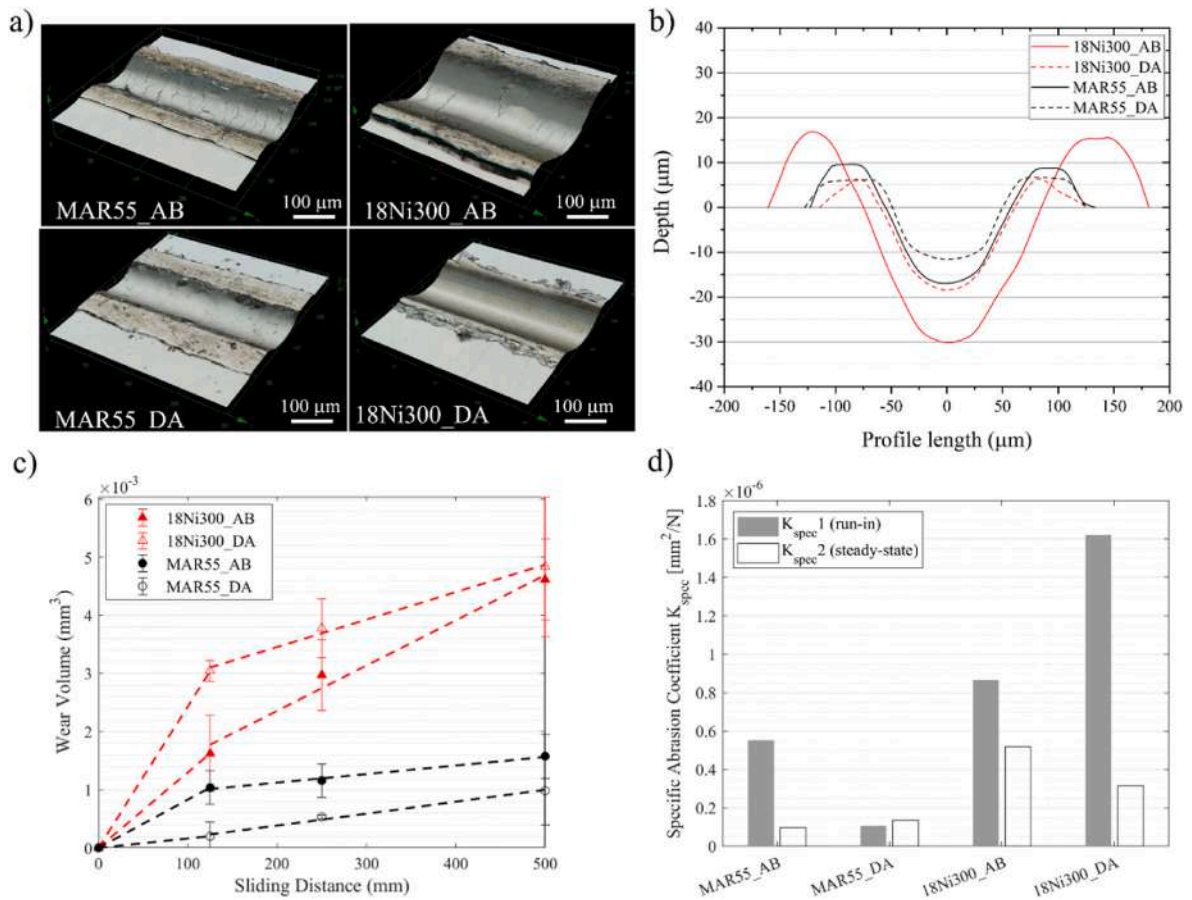


Fig. 10. Scratch test results for additively manufactured MAR55 and 18Ni300 alloys in AB and DA conditions, showing (a) 3D profilometry and (b) cross-sectional profiles after 100 cycles (500 mm sliding distance), (c) wear volume versus sliding distance, and (d) specific abrasive coefficients (K_{spec}) for the run-in and steady-state stages.

conductivity values slightly below those of the wrought H13 quenched and tempered alloy.

Overall, thermal conductivity follows an increasing trend from 18Ni300 DA to MAR55 AB, then to additively manufactured H13 in the directly tempered condition, followed by MAR55 DA, with wrought and quench and tempered H13 exhibiting the highest values across the entire investigated temperature range. It should be noted that the reference H13 (wrought QT and AM DT) shows ultimate tensile strengths of 1900–2000 MPa, compared to MAR55, which ranges from ~1600 MPa in the AB condition to ~1900 MPa in the DA condition at peak hardness.

Fig. 10 presents the scratch test results for additively manufactured MAR55 and 18Ni300 alloys in the AB and DA conditions. The 3D profilometry and cross-sectional profiles of the wear tracks after 100 cycles are shown in Fig. 10a and b, respectively. The wear volumes at 25, 50, and 100 cycles are plotted in Fig. 10c, and the corresponding abrasive wear coefficients for the initial run-in and steady-state stages are shown in Fig. 10d.

Table 4 summarizes the hardness of the tested materials, along with wear test results, including wear rates and specific abrasive coefficients for the run-in stage (0–25 cycles, corresponding to sliding distances up to 125 mm) and the steady-state stage (25–100 cycles, corresponding to sliding distances from 125 to 500 mm).

The wear behavior of additively manufactured MAR55 and 18Ni300 alloys in AB and DA conditions was evaluated in terms of wear rate (WR) and specific abrasive coefficient (K_{spec}), calculated according to Archard's wear law considering the wear volume, the sliding distance, the applied load and material hardness [41].

MAR55 AB shows lower abrasive wear than 18Ni300 in both run-in and steady-state stages. During run-in, the condition exhibits a higher wear rate ($8.29 \times 10^{-6} \text{ mm}^3/\text{mm}$) than DA ($1.60 \times 10^{-6} \text{ mm}^3/\text{mm}$), consistent with its lower hardness ($471 \pm 12 \text{ HV10}$ vs $542 \pm 10 \text{ HV10}$). The specific abrasive wear coefficient K_{spec} reflects this difference, with AB reaching $0.55 \times 10^{-6} \text{ mm}^2/\text{N}$ and DA only $0.11 \times 10^{-6} \text{ mm}^2/\text{N}$. This indicates that the softer AB material is more susceptible to material removal per unit load. In the steady-state regime, AB shows a decreased wear rate ($1.47 \times 10^{-6} \text{ mm}^3/\text{mm}$), corresponding to a lower K_{spec} of $0.10 \times 10^{-6} \text{ mm}^2/\text{N}$, while DA experiences a slight increase in wear rate ($2.04 \times 10^{-6} \text{ mm}^3/\text{mm}$), resulting in K_{spec} of $0.14 \times 10^{-6} \text{ mm}^2/\text{N}$. These trends suggest a transition from a run-in stage dominated by plastic deformation in AB to a steady-state regime where abrasive wear is more controlled by hardness. The prevalence of plastic deformation cracks in AB after 100 cycles further supports the higher plastic deformation contribution in AB rather than DA (Fig. 10a).

For 18Ni300, during the run-in stage, the more ductile AB material ($336 \pm 4 \text{ HV10}$) accommodates plastic deformation through pronounced lateral pile-up, forming deep wear tracks but a relatively low total wear volume, as most of the material is displaced rather than removed. This is evidenced by the deformation cracks on the wear track

(Fig. 10a), the high penetration depth, and the pronounced pile-up observed after 100 cycles (Fig. 10b). As a result of its low hardness, limited abrasive contribution, and predominance of plastic deformation, which reduces the effective abrasive wear rate, the AB condition exhibits a K_{spec} of $0.87 \times 10^{-6} \text{ mm}^2/\text{N}$.

The harder DA condition ($598 \pm 7 \text{ HV10}$), by contrast, undergoes more direct abrasive removal, exhibiting a higher run-in K_{spec} of $1.62 \times 10^{-6} \text{ mm}^2/\text{N}$ compared to AB ($0.87 \times 10^{-6} \text{ mm}^2/\text{N}$), as abrasion dominates in DA while plastic deformation prevails in AB. In the steady-state regime, wear rates decrease for both conditions, but now abrasive wear governs in both materials. Consequently, AB reaches a steady-state K_{spec} of $0.52 \times 10^{-6} \text{ mm}^2/\text{N}$, while DA decreases further to $0.31 \times 10^{-6} \text{ mm}^2/\text{N}$, reflecting the higher hardness and improved resistance to abrasion. The observed wear mechanisms, predominantly plastic deformation in AB during run-in and dominant abrasion in DA, are consistent with surface morphologies after 100 cycles, showing deep pile-up in AB and cleaner, well-defined wear tracks in DA (Fig. 10a and b). These macroscopic observations are further supported by Supplementary File 2, which provides SEM-scale evidence of the wear mechanisms. In particular, the SEM analysis confirms the transition from plasticity-dominated wear in the AB condition to a stable abrasive regime after DA, with complete suppression of plastic cracking in both alloys. In addition, MAR55 DA exhibits a narrower wear track than 18Ni300 DA, confirming its higher resistance to material removal despite its slightly lower hardness (~550 HV10 vs ~600 HV10). This indicates that a Fe–C martensitic matrix strengthened by Mo₂C precipitation provides more effective resistance to abrasive micro-cutting than a softer Fe–Ni martensite strengthened by Ni₃Ti intermetallics in 18Ni300, thereby limiting overall wear damage.

3.4. Ageing (tempering) kinetics

To elucidate the mechanisms underlying ageing (tempering) in MAR55, a systematic study was performed using dilatometry (Dil.) and DSC. A comprehensive understanding of the ageing mechanisms is essential, as it provides a framework for interpreting the observed microstructural evolution and the corresponding mechanical and thermal properties presented in the previous section.

Fig. 11 presents the heat flux (red curve) and the first derivative of the relative length change with respect to temperature, $d(\Delta l/l_0)/dT$ (black curve), at a heating rate of $2^\circ\text{C}/\text{min}$. The derivative is plotted instead of the dimensional change in length because it is more sensitive

Table 4
Hardness of the tested materials, wear rate and wear abrasion coefficient of the run-in and steady state stage.

Material	Hardness (HV10)	Wear rate $\times 10^{-6} \text{ (mm}^3/\text{mm)}$		Specific abrasion coefficient $\times 10^{-6} \text{ (mm}^2/\text{N)}$	
		WR1 run-in	WR2 steady state	K_{spec1} run-in	K_{spec2} steady state
MAR55 AB	471 ± 12	8.29	1.47 ($R^2 = 0.986$)	0.55	0.10
MAR55 DA	542 ± 10	1.60	2.04 ($R^2 = 0.989$)	0.11	0.14
18Ni300 AB	336 ± 4	12.98	7.78 ($R^2 = 0.982$)	0.87	0.52
18Ni300 DA	598 ± 7	24.32	4.72 ($R^2 = 0.992$)	1.62	0.31

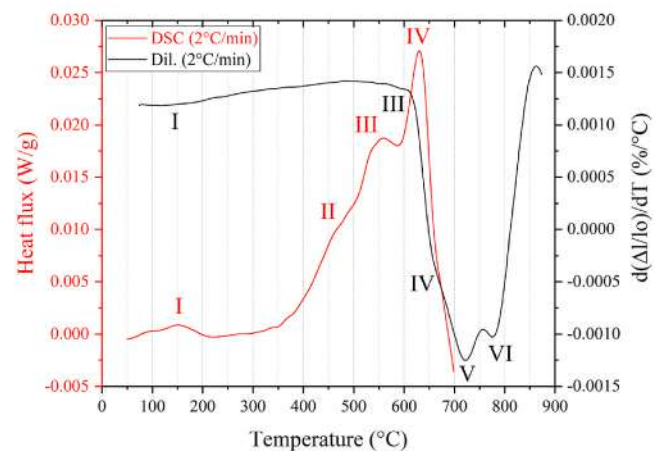


Fig. 11. Heat flux (red curve) and the first derivative of the relative change in length with respect to temperature, $d(\Delta l/l_0)/dT$ (black curve), measured by dilatometry acquired with a heating rate of $2^\circ\text{C}/\text{min}$. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

to small volume changes.

The slow heating rate of 2 °C/min was selected to highlight the precipitation phenomenon occurring before austenite reversion during heating [58,59].

To further investigate the ageing behavior of MAR55, additional DSC and dilatometric measurements were performed at various heating rates. The corresponding data are reported in the Supplementary File 2 and were used to calculate the activation energies following the Arrhenius-like Kissinger equation (Eq. (3)) [45,46]. The resulting activation energies, together with the corresponding adjusted R^2 values and the associated phase transformations, are summarized in Table 5.

To evaluate the thermal stability of MAR55 under service-relevant conditions, its behavior at 600°C was specifically examined. This temperature range corresponds to typical operating conditions for hot-work tool steels but is generally unsuitable for conventional maraging steels due to the onset of austenite reversion. The dilatometric curve obtained during isothermal ageing at 600°C for 40 h is shown in Fig. 12. Subsequently, an isochronal heating up to 900°C at a rate of 1°C/min was performed on the overaged sample, and the resulting curve was compared with that of the AB material (Fig. 12b).

A contraction is observed at the beginning of the isothermal stage, followed by a slight additional contraction between 37 h and 40 h of dwell time and an expansion during cooling, occurring around 200°C (Fig. 12a). Comparing the two curves in Fig. 12b, the disappearance of peaks III and IV after 40 h overageing at 600°C is evident. This indicates that the diffusion-controlled transformations responsible for these two peaks occur during the 600°C long time soaking, as demonstrated by the contraction observed during the isothermal stage (Fig. 12a).

Light optical microscopy (LOM) after etching with Lichtenegger and Bloch's colored reagent, along with scanning electron microscopy (SEM) micrographs, of samples aged at 600°C for up to 40 h, are presented in Fig. 13.

Etching with Lichtenegger and Bloch's reagent clearly shows that the cellular solidification substructure persists up to 20 h of ageing at 600°C, whereas its complete disappearance is observed only after 40 h (LOM micrographs in Fig. 13). SEM micrographs acquired in backscattered electron mode reveal chemical inhomogeneity, with darker cellular regions and lighter intercellular areas, indicating compositional variation. EDXS analysis was performed on these regions, denoted as Spot 1 (darker) and Spot 2 (lighter) in Fig. 13, and used to calculate the residual segregation index (I_{RS}) at different dwell times at 600°C, according to Eq. (4) [60].

$$I_{RS} = \frac{C_{max}^t - C_{min}^t}{C_{max}^0 - C_{min}^0} \quad \text{Eq. 4}$$

Table 5
Summary of phase transformations in MAR55 detected by dilatometry (Dil.) and DSC during heating up to 900°C.

Peak	Technique	Temperature range (°C)	Activation energy (kJ·mol ⁻¹)	Adj. R ² (–)	Transformation
I	Dil. and DSC	100–200	93.5 (Dil.) 99.5 (DSC)	0.955 0.991	Clustering and/or precipitation of transition carbides
II	DSC	450–500	170.8 (DSC)	0.985	$\gamma \rightarrow \alpha + \text{Fe}_3\text{C}$
III	Dil. and DSC	500–600	233.8 (Dil.) 214.6 (DSC)	0.971 0.993	Precipitation of alloyed Fe ₃ C
IV	Dil. and DSC	600–650	256.1 (Dil.) 282.1 (DSC)	0.962 0.995	$\gamma_{(1 \text{ el.}\%)} \rightarrow \gamma_{(1 \text{ el.}\%)} + \text{Mo}_2\text{C}$
V	Dil.	650–750	243.7 (Dil.)	0.979	γ reversion via Ni diffusion
VI	Dil.	>750 (up to 900)	824.1 (Dil.)	0.976	Displacive γ reversion

Here, C corresponds to the amount of the element in the rich area (C_{max}) and in the lean area (C_{min}), t represents the dwell time under consideration, and 0 denotes the initial condition, specifically AB. The full EDXS datasets used for this analysis are provided in the Supplementary File 3. The I_{RS} values for Ni, Co, Mo, and Cr at various times at 600°C are shown in Fig. 14. The I_{RS} for Si was not calculated due to its low concentration in all conditions lower than 1 wt%.

The Ni I_{RS} demonstrates an increase over a period of up to 20 h, after which it stabilizes, in contrast to the other elements, which exhibit no significant variation during ageing at 600°C.

The evolution of RA was analyzed by ex-situ XRD after electropolishing on samples treated at 480°C for 1 h, and at 600°C for 1, 4, and 40 h, followed by quenching. Complete XRD plots, including spectra plotted with the squared intensity to highlight small peaks, are provided in Supplementary File 4. Quantitative analysis, performed using a combined Rietveld method via MAUD software, is summarized in Table 6 [51].

A decrease in the amount of RA is detected after treatment at 480°C for 1 h. Upon tempering at 600°C for 1 h, a slight decrease in austenite is observed compared to the AB condition. After a prolonged dwell time of 4 h at 600°C, an increase in austenite occurs, followed by a decrease after a dwell time of 40 h. The austenite content measured after tempering at 600°C for 40 h was approximately 16 vol%. For comparison, Conde et al. [44] reported an austenite content of 48 vol% after tempering at 610°C for only 40 min for a 18Ni300 maraging steel. These values align with the calculated equilibrium austenite content at approximately 600°C for MAR55 and 18Ni300, which are 20 vol% and 50 vol%, respectively (Supplementary File 1). The slight discrepancies between the calculated equilibrium values and the experimental measurements can be attributed to a certain amount of martensitic transformation that occurs during cooling. To elucidate the reasons for the variations in austenite content with increasing tempering temperature, the cooling dilatometric curves after treatment at 480°C and 600°C for 1 h were compared, as shown in Fig. 15a. Additionally, to investigate the effects of different dwell times at 600°C, the cooling curves after 1, 4, and 40 h are presented in Fig. 15b.

Evaluation of cooling dilatometric curves after different tempering temperatures, specifically 480°C (~near the peak ageing temperature) and 600°C (~ temperature at which austenite reversion kinetics is high), with equal dwell times of 1 h, can highlight the occurrence of martensitic transformation, as shown in Fig. 15a.

The coefficient of thermal expansion (CTE) of the AB material in the 400–500°C range is $13.9 \times 10^{-6} \text{ °C}^{-1}$, as determined from the dilatometry curve shown in Fig. 12b. No variation in the CTE is observed during cooling after tempering at 480°C for 1 h. A martensitic transformation with an Ms temperature close to 160°C is evident. Comparing the cooling curves after tempering at 480°C for 1 h (black curve in Figs. 15a) and 600°C for 1 h (red curve in Fig. 15a), an increase in both the Ms temperature and CTE with increasing tempering temperature is detected. Analysis of the cooling dilatometric curves following various dwell times at 600°C reveals a further increase in the CTE after 4 h at 600°C (Fig. 15b) and a decrease in the Ms temperature. Extending the dwell time to 40 h (red curve in Fig. 15b) results in a slight increase in the Ms temperature and a continued increase in CTE.

In-situ synchrotron XRD experiments were performed on polished samples to monitor the evolution of the austenite phase during heating of both MAR55 and 18Ni300 alloys, with spectra collected at 200, 380, 450, 480, 500, 525, 550, 575, 600, 626, and 650°C. The austenite amount (γ , vol%) and the austenite lattice parameter (a_γ) as a function of temperature, evaluated using MAUD software [51], for both MAR55 and 18Ni300 are reported in Fig. 16.

At the beginning of heating, the MAR55 alloy contains approximately 10 vol% austenite, about 5 vol% lower than the value measured after electropolishing (Table 6), whereas 18Ni300 contains only ~1 vol % retained austenite. Upon heating, both alloys exhibit an increase in the austenite fraction due to diffusion-controlled reversion. In 18Ni300,

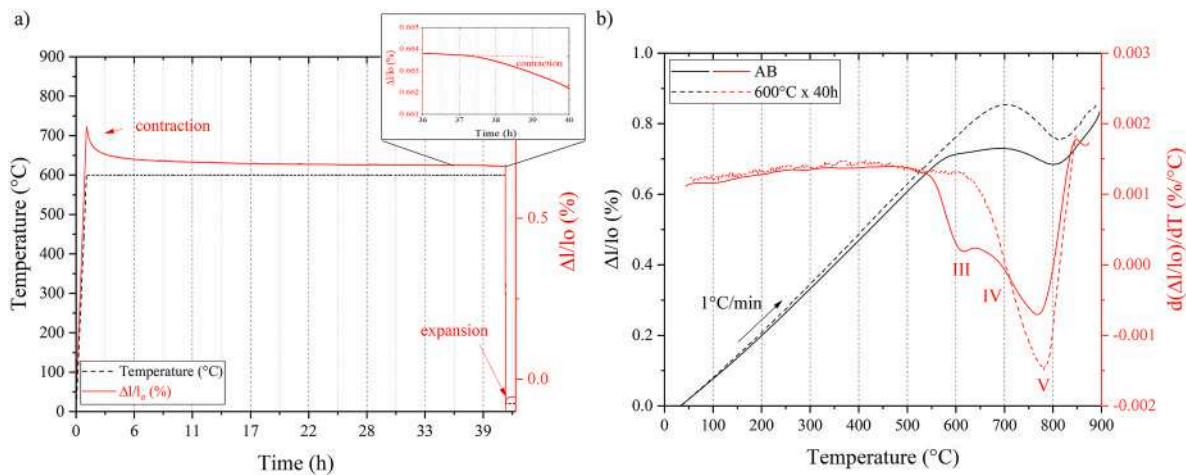


Fig. 12. a) Dilatometric curve of the heat treatment at 600°C for 40 h followed by a quench at 100 °C/s b) Change in length (black curves) and its derivative with respect to temperature (red curves) during isochronal heating at 1 °C/min up to 900°C for the AB sample and the sample treated at 600°C for 40 h. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

reversion initiates at around 480°C and continues up to 650°C, reaching approximately 40 vol% austenite. In MAR55, the onset of reversion is delayed to around 600°C, and the fraction increases more modestly to about 20 vol% at 650°C. Considering the initial austenite content, the net increase amounts to roughly 39 vol% in 18Ni300 and only about 10 vol% in MAR55, indicating that austenite formation during heating is significantly more pronounced in 18Ni300. This behavior reflects the earlier and more extensive reversion promoted by its higher Ni content and the lower thermodynamic barrier for γ -phase formation. In contrast, the presence of Mo and reduced Ni diffusivity in MAR55 hinders reversion at intermediate temperatures.

The lattice parameter evolution (bottom graph in Fig. 16) provides complementary insight into the underlying thermodynamic and compositional processes. Up to approximately 400°C, a_γ in MAR55 increases linearly, as expected from thermal expansion of the fcc lattice. Between 450°C and 550°C, a distinct contraction is observed in MAR55, indicating solute depletion, from the austenite matrix due to carbide precipitation as commonly observed in tool steels [61]. For 18Ni300, the lattice parameter is not reported below 525°C due to the very low austenite fraction (~1 vol%), which makes reliable lattice parameter evaluation with MAUD challenging. The lattice parameter could only be accurately measured once the austenite peak became well-defined, which occurs above 525°C. Then, for both MAR55 and 18Ni300, an increase in the lattice parameter is observed above 575°C, which is likely related to reversion (i.e., enrichment in Ni) as also indicated by Thermo-Calc predictions. The increase is much more pronounced in 18Ni300 due to its higher Ni content, reflecting faster reversion kinetics and the stronger influence of Ni on the lattice parameter.

The occurrence of austenite reversion in MAR55 at higher temperatures compared to 18Ni300 is evident through dilatometric analysis reported in Fig. 17. Fig. 17 clearly illustrates the three contractions associated with the precipitation phenomenon, austenite reversion driven by diffusion, and austenite transformation induced by shear in 18Ni300 [62]. In contrast, MAR55 exhibits a distinct shift of these phenomena toward higher temperatures, which explains the onset of the increase in austenite content at elevated temperatures, as shown in Fig. 17.

4. Discussion

4.1. Microstructure and properties of MAR55 in as-built state

The L-PBF processed MAR55 alloy in the AB condition shows a hardness of about 490 HV10, an ultimate tensile strength above

1600 MPa, and a Charpy impact energy close to 60 J. Its microstructure is mainly composed of martensite together with approximately 15 vol% of intercellular RA. The microstructural features of the MAR55 AB alloy are governed by the off-equilibrium solidification conditions inherent to the L-PBF process [1]. Scheil solidification modelling (Fig. 3a) reveals pronounced Mo and C segregation in the last liquid to solidify along cellular boundaries, resulting in strong intercellular microsegregation. This chemical partitioning locally suppresses the martensite start (M_s) temperature to near-ambient levels, thereby stabilizing retained austenite within solute-enriched regions. Such segregation is a direct consequence of the high cooling rates and constitutional undercooling associated with L-PBF processing, which simultaneously promote martensitic transformation and solute redistribution at the cellular scale. The high thermal gradients also promote a refined hierarchical structure of lath martensite within the prior austenite grains. A comparison with 18Ni300 maraging steel in the AB state highlights the distinct mechanical response of MAR55. In particular, MAR55 exhibits a yield strength about 27% higher (1496 ± 41 MPa compared to 1179 ± 19 MPa) and an ultimate tensile strength about 37% higher (1626 ± 7 MPa compared to 1189 ± 2 MPa), while maintaining a very similar elongation to failure ($17.9 \pm 0.3\%$ versus $18.2 \pm 0.1\%$). On the other hand, the impact toughness is significantly lower, showing a reduction of approximately 46% (58 ± 1 J compared to 108 ± 3 J). These differences can be mainly attributed to the presence of carbon-strengthened martensite in MAR55, whereas 18Ni300 is characterized by a softer carbon-free Fe-Ni martensitic matrix containing only limited amounts of retained austenite. The enhanced tensile performance is further reflected in the derived strength-ductility balance and tensile toughness parameters, which both confirm the superior mechanical efficiency of MAR55 in the AB condition compared to the 18Ni300 AB benchmark. The combination of these features explains why the alloy, despite its very high strength, is still able to retain good ductility, with elongation values close to 18%, together with relatively high impact toughness in the AB state. The mechanical response of the MAR55 AB alloy is strongly governed by the heterogeneous cellular microstructure generated during L-PBF processing. The C- and Mo-enriched intercellular regions stabilize approximately 15 vol% retained austenite, whereas the cellular cores transform into a hard lath martensitic matrix. This heterogeneous architecture creates a composite-like deformation behavior during tensile loading, where the softer retained austenite locally accommodates plastic strain while the martensitic matrix provides high load-bearing capacity. The progressive strain partitioning between these constituents delays strain localization and promotes sustained work hardening, explaining the unusual

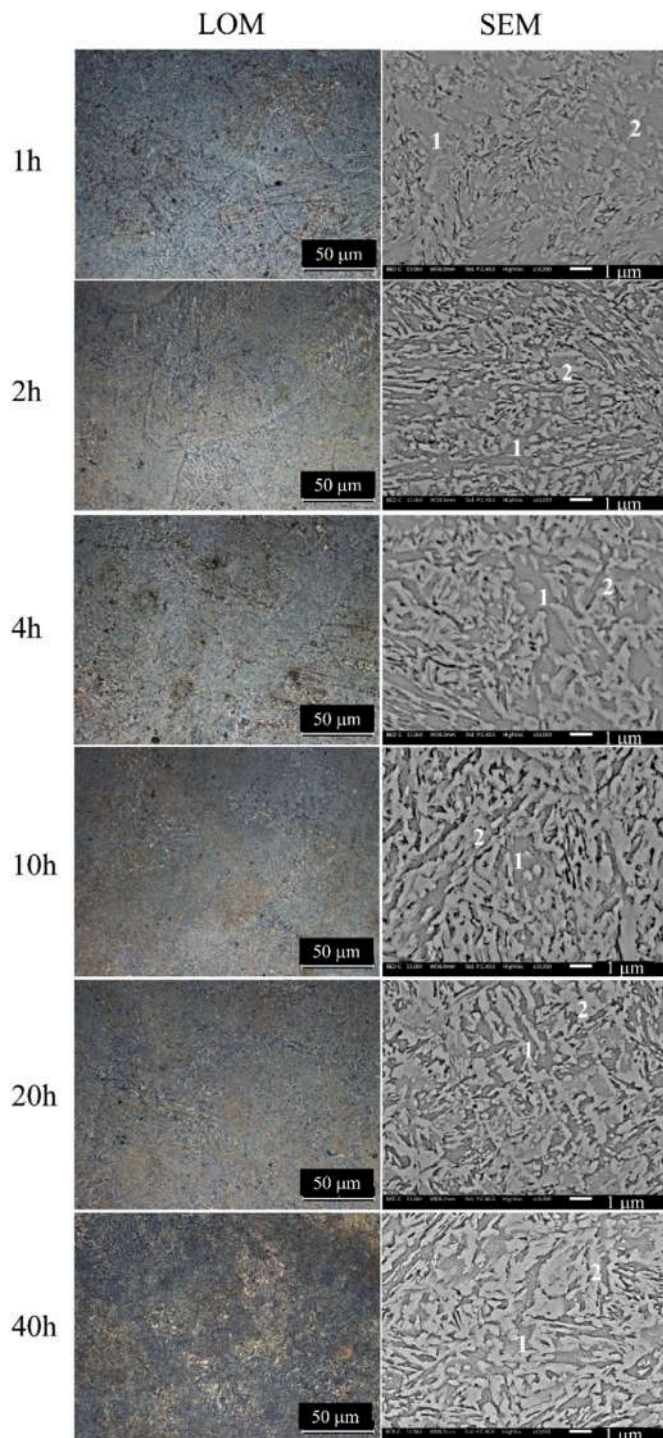


Fig. 13. Micrographs of MAR55 samples aged at 600°C for 1, 2, 4, 10, 20, and 40 h, etched using Lichtenegger and Bloch's reagent, acquired by optical microscopy (LOM) and backscattered electron imaging in SEM.

combination of very high strength (>1600 MPa) and relatively high elongation (~18%). Under quasi-static tensile loading, the presence of intercellular retained austenite promotes strain partitioning and enhances work hardening, thereby increasing both the strength–ductility synergy and the overall energy absorption capacity up to fracture. By contrast, under dynamic loading conditions such as Charpy impact testing, the strong local mechanical mismatch between hard martensite and retained austenite-rich intercellular regions promotes local stress concentration at phase boundaries, facilitating earlier crack initiation

and propagation. This explains why, despite excellent tensile toughness indicators, MAR55 exhibits lower impact toughness compared to 18Ni300.

4.2. Study of ageing through thermal analyses

Based on dilatometric and DSC analyses, six distinct phase transformations occur during heating of MAR55 up to 900°C. The first transformation (100–200°C) involves the clustering and/or precipitation of transition carbides [40]. Although the nominal carbon content of MAR55 is below 0.20 wt%, the presence of localized C-rich micro segregated regions (Fig. 3), may locally provide sufficiently high carbon concentrations to promote the precipitation of transition carbides, thus accounting for the observed transformation peak. This transformation is not observed in Fe–C martensitic tool steels produced by L–PBF when a preheated build plate is used, due to the intrinsic tempering effect induced by the process [17]. While clearly detected by DSC, it is less evident in dilatometry due to the low transformation strain. Activation energies of 93.5 kJ/mol (dilatometry) and 99.5 kJ/mol (DSC) align with literature values (80–111 kJ/mol) [63].

The second phenomenon, occurring in the temperature range of 450–500°C, is detected only in the DSC signal and is not observable in the dilatometric curves. It is characterized by an activation energy of 170.8 kJ/mol, which closely matches the reported range for the decomposition of a small amount of retained austenite into ferrite and cementite (166–185 kJ/mol) [40,64]. This transformation is unlikely to produce a noticeable signal in dilatometry, suggesting that only a small fraction of RA is involved. However, the highly alloyed austenite can experience chemical dilution during heating due to carbide precipitation, which may facilitate martensitic transformation during subsequent cooling, as observed in the specimen tempered at 480°C. Most of the retained austenite present in the AB material remains stable at this temperature, as confirmed by the martensitic transformation seen after tempering at 480°C and 600°C (Fig. 15a), and by in-situ synchrotron XRD analysis, which shows no significant change in austenite content until 600°C (Fig. 16). This behavior is further illustrated by the reduction in the lattice parameters of austenite measured in the synchrotron experiments, reflecting the effects of chemical dilution.

The third transformation, occurring around 500–600°C, corresponds to alloyed cementite precipitation. This is supported by the measured activation energies of 233.8 kJ/mol from dilatometry and 214.6 kJ/mol from DSC [40,46].

The fourth phenomenon evident in both thermal analyses, occurring at around 600–650°C, corresponds to the precipitation of Mo-rich phases [37,43,59,65,66]. In-situ synchrotron XRD reveals a lattice contraction in MAR55 between 450 and 550°C (Fig. 16), consistent with C and Mo depletion from the austenite matrix due to Mo₂C precipitation, as demonstrated by Paul A Davies et al. [53]. The contraction reflects lattice shrinkage following austenite decomposition. This finding is further supported by the measured activation energies of 256.1 kJ/mol (dilatometry) and 282.1 kJ/mol (DSC), which are in good agreement with the activation energies associated with the diffusion of substitutional elements in ferrite, specifically 245.8 kJ/mol for Ni, 272 kJ/mol for Ti, and 251 kJ/mol for Mo [67–69]. At these temperatures, the precipitation of Mo₂C reduces the local stability of retained austenite, particularly in the intercellular regions, promoting martensitic transformation during subsequent cooling. This behavior aligns with the observed Ms during cooling after tempering at 480°C for 1 h (Fig. 15a), showing that, although the overall austenite fraction remains largely unchanged during the isothermal treatment, the intercellular retained austenite becomes susceptible to martensitic transformation due to local depletion of alloying elements. As a result, the final retained austenite content is lower than in the as-built state, reflecting the combined effects of early carbide precipitation and martensitic transformation during cooling. This observation explains the reduction in the final retained austenite content after ageing at 480°C for 1 h, as compared to the

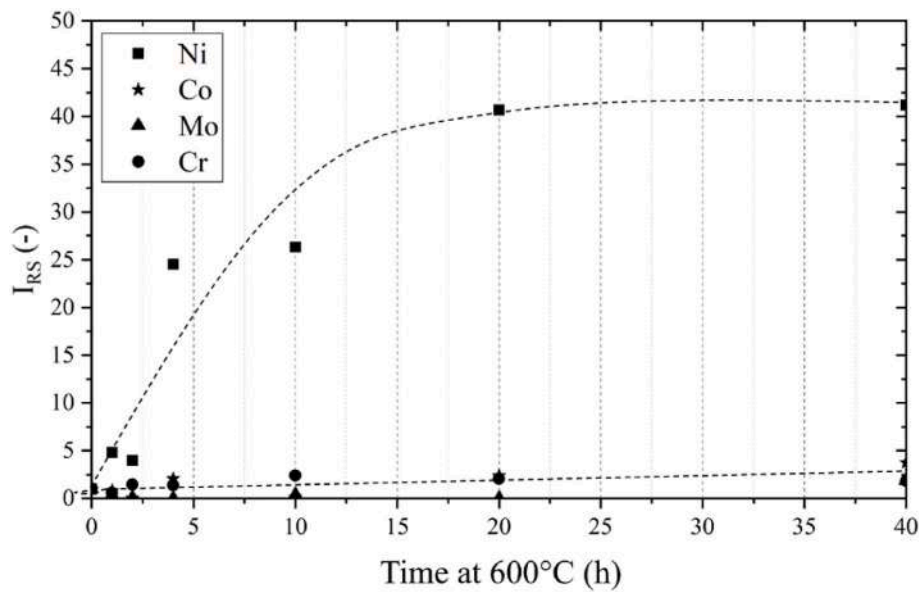


Fig. 14. Evolution of the residual segregation index of Ni, Co, Mo and Cr versus time during the isothermal treatment at 600°C.

Table 6

Quantitative analysis achieved by using a combined Rietveld method via MAUD software.

	α'/α (vol%)	γ (vol%)
AB	85	15
480°C 1h	97	3
600°C 1h	87	12
600°C 4h	79	20
600°C 40h	83	16

as-built state, decreasing from 15 vol% to 3 vol% (Table 6).

The fifth transformation detected through dilatometry is related to the reversion of austenite. This is driven by the diffusion of austenite-stabilizing elements towards the interfaces between martensite laths and prior austenite grain boundaries, within a compositionally homogenized martensitic structure [43,59,67]. In contrast, within non-homogenized matrices, localized reversion tends to occur preferentially at regions with elevated concentrations of austenite-stabilizing elements [70]. A.L. M. Feitosa et al. [70] investigated the relationship between the

precipitation of strengthening phases and austenite reversion in Maraging 350 steel, noting that at higher temperatures, the intermetallic precipitates, specifically $\text{Ni}_3(\text{Ti},\text{Mo})$ and Fe_2Mo , coalesced and partially dissolved. MAR55 behaves differently due to the higher thermal stability of Mo_2C . Unlike the intermetallics in Maraging 350, Mo_2C remains stable and does not dissolve prior to austenite reversion, thus no additional elements are released to promote austenite growth. XRD analysis (Table 6 and Supplementary File 4) confirms the persistence of Mo_2C after 40 h at 600°C, demonstrating its stability and lack of contribution to the onset of austenite reversion.

The activation energy for austenite reversion via diffusion, measured in MAR55 as 243.7 kJ/mol, is consistent with the reported values for the lattice diffusion of Ni in iron (245.8 kJ/mol) [67].

The residual segregation index measured after different isothermal times (Fig. 14) provides clear evidence that Ni diffusion is the primary mechanism facilitating austenite reversion [43,59,67]. In addition, at 600°C, changes in the coefficient of thermal expansion (CTE) provide clear indirect evidence of austenite reversion. After 1 h of tempering, an increase in the CTE compared to the as-built condition indicates the early formation of austenite, likely concurrent with or following the

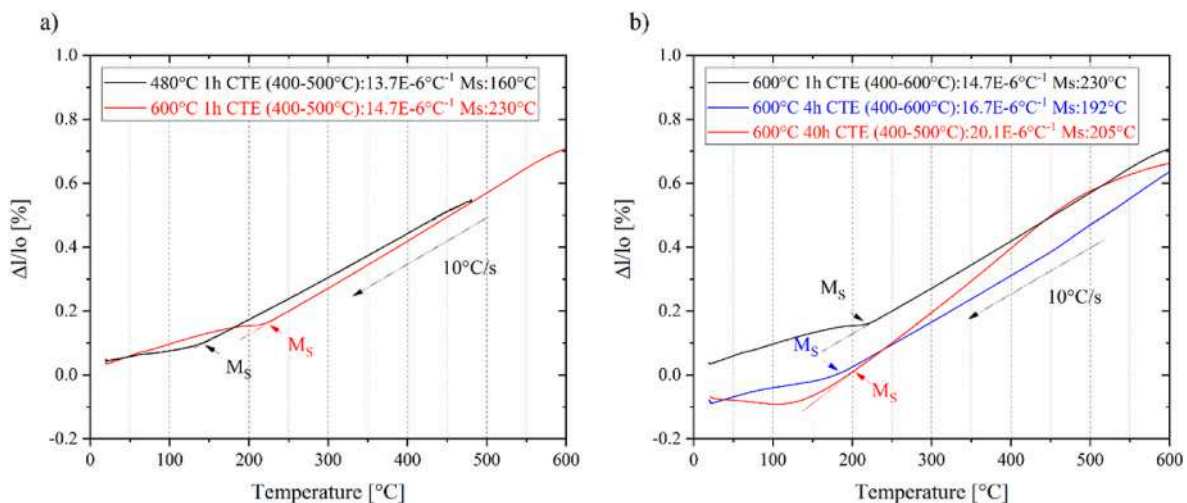


Fig. 15. a) The cooling dilatometric curves after different tempering temperatures, namely 480°C and 600°C, for 1 h. b) The cooling dilatometric curves after different dwell times at 600°C, namely 1, 4, and 40 h.

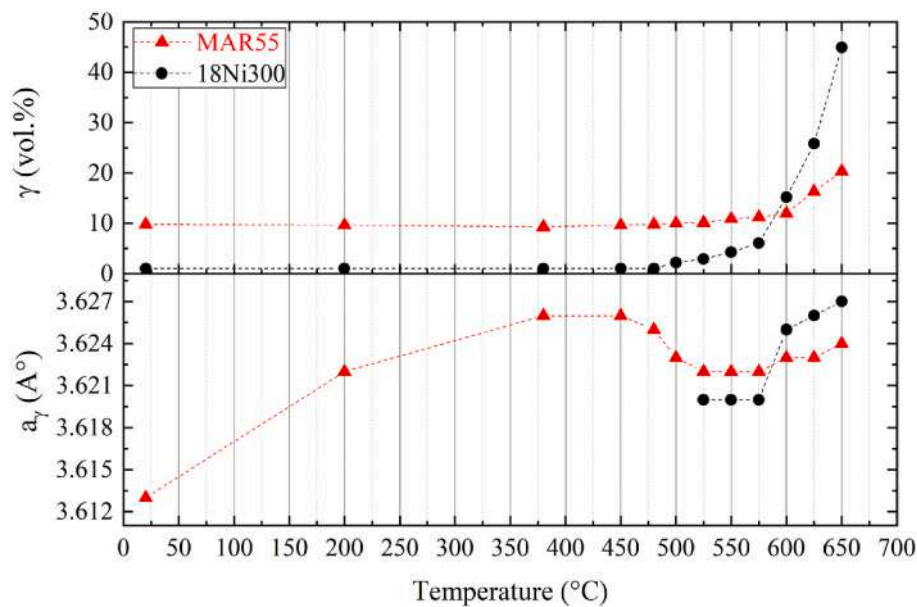


Fig. 16. Evolution of the retained austenite fraction (γ , top) and austenite lattice parameter (a_γ , bottom) as a function of temperature for 18Ni300 (black circles) and MAR55 (red triangles), obtained from in-situ synchrotron XRD measurements during heating. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

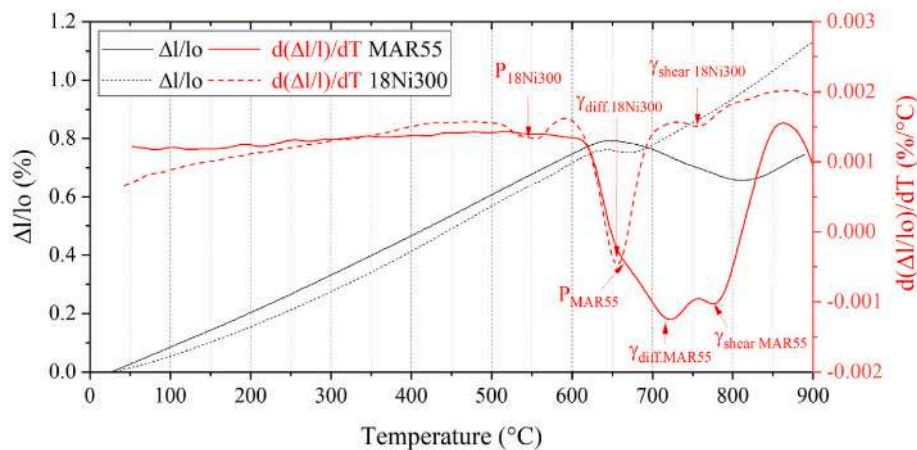


Fig. 17. Change in length (black curves) and its derivative with respect to temperature (red curves) during isochronal heating at 10 °C/min up to 900°C for the MAR55 and 18Ni300. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

precipitation of Mo_2C . Since the CTE of austenite is higher than that of martensite ($\text{CTE } \gamma > \text{CTE } \alpha^1$), this increase reflects the growing austenitic fraction within the microstructure [71,72]. Prolonged holding at 600°C (4 h and 40 h) results in a progressive increase in CTE, confirming continued austenite reversion as nickel diffusion promotes chemical homogenization. The slight decrease in M_s temperature after 4 h suggests that the newly formed austenite becomes increasingly stabilized by Ni enrichment, while the small increase in M_s observed after 40 h may indicate a re-partitioning of alloying elements and partial homogenization between the γ and α^1 phases. This trend is further corroborated by the analysis of the cooling dilatometric curves (Fig. 15a and b), which reveal the evolution of M_s temperature as a function of dwell time at 600°C. After 1 h, the higher M_s (~230°C) indicates reduced austenite stability due to carbide precipitation, while after 4 h, Ni diffusion promotes the formation of more stable austenite with a lower M_s (~190°C). After 40 h, a slight increase in M_s suggests partial homogenization and redistribution of Ni between γ and α^1 phases. Overall, the combined evolution of CTE and M_s temperature during tempering reveals that austenite reversion proceeds through a Ni diffusion-controlled

mechanism. Compared to 18Ni300, where reversion starts at lower temperatures (~450°C) and results in higher austenite fractions (~40 vol% at 650°C), the studied alloy exhibits slower reversion kinetics, highlighting the strong influence of chemical composition on transformation behavior.

As the temperature increases, thermodynamic forces become more prominent, leading to the sixth transformation detected in MAR55, associated with the transformation of the remaining martensite to austenite via a shear mechanism (peak VI). In all conditions tested, where the heating rate is below 55 °C/s, sufficient time is available for diffusion, resulting in the observation of both peaks at each heating rate (Supplementary File 2) [59,73]. Consequently, the sixth transformation phenomenon involves the shear transformation of the remaining martensite (α^1) to austenite (γ). The activation energy for this process is determined to be 824.1 kJ/mol with an adjusted R-squared value of 0.976, significantly higher than the activation energies associated with the diffusion-dominated transformations. The higher activation energies, compared to those for austenite reversion via diffusion, are consistent with a different mechanism, namely shear rather than

diffusion. However, the literature does not always distinguish between these two contributions, resulting in a wide range of activation energy values reported for austenite reversion [42,44,59,62,67,74]. Moreover, there is variation in the activation energy for martensite reversion depending on the heating rate. Kapoor et al. [59] found that for heating rates below 2°C/s, the activation energy for martensite reversion is 423 kJ/mol. For heating rates above 2°C/s, the activation energy increases to 828 kJ/mol, which corresponds to the value observed for MAR55, although at lower heating rates. Activation energies of approximately 562 kJ/mol and 224 kJ/mol for austenite reversion were reported by Carvalho et al. [42] and Viswanathan et al. [74], respectively, while Guo et al. [67] and Kröl et al. [62] reported values of 342 kJ/mol and 478 kJ/mol, respectively, for a range of maraging steels, including 18Ni250, 18Ni300, and 18Ni350. This increase in activation energy is attributed to the more pronounced shear mechanism at higher heating rates.

The microstructural evidence of increased retained austenite, together with thermal observations, indicates that prolonged exposure at 600°C may involve the onset of shear-assisted transformation of martensite into austenite. This is further supported by the slight contraction observed between 37 and 40 h of dwell time, providing additional evidence for the shear-assisted martensite-to-austenite transformation and complementing the Ni diffusion-driven austenite reversion that occurs during the earlier stages of the treatment.

A schematization of the sequence of phenomena occurring during tempering at 600°C for 40 h is reported in Fig. 18, showing the evolution of hardness, dimensional change, Ni residual segregation index, and retained austenite content as determined from ex-situ XRD.

In summary, the phase transformations occurring during the tempering of L-PBFeD MAR 55, dependent on tempering temperature and time, can be delineated as follows: tempering at 480°C for 1–2 h initially induces the precipitation of Mo₂C carbides and the decomposition of intercellular RA, resulting in peak ageing of the alloy similar to those of C bearing L-PBF tool steels. Increasing the ageing temperature to 600°C facilitates the diffusion of Ni, promoting the reversion of austenite similar to that of C-free maraging steels. This reversion process, along with the continued coarsening of strengthening carbides,

leads to a reduction in hardness.

4.3. Microstructure and properties of aged MAR55

The direct ageing of MAR55 was carried out at 480°C, a temperature selected based on thermal analysis as promoting Mo₂C precipitation without austenite reversion, thus maximizing precipitation strengthening within the martensitic matrix [53]. Microstructural characterization confirms that the hierarchical lath-block-packet morphology inherited from the prior austenite grains is largely preserved. However, the isothermal treatment induces variant selection and partial recovery within the martensitic laths, resulting in slightly coarser packets with more uniform crystallographic orientations. A concurrent and significant reduction in retained austenite (RA) is observed, particularly along the intercellular boundaries. This suggests that the intercellular RA, which is stabilized by micro-segregation in the AB state, becomes thermodynamically unstable as it undergoes solute depletion (specifically of C and Mo) during the formation of carbides, subsequently transforming into martensite during cooling.

The transition from the AB to the DA state can be explained through a sequence of thermally activated microstructural transformations occurring during ageing. Isothermal holding at 480°C primarily induces the precipitation of fine Mo₂C carbides (Peak IV), as evidenced by the reported activation energy (256.1 kJ/mol) and the austenite lattice contraction observed via in-situ synchrotron analysis. This precipitation induces a progressive chemical depletion of Mo and C from the intercellular retained austenite, thereby reducing its thermodynamic stability. As a consequence, this austenite becomes metastable and partially transforms into martensite during subsequent cooling, leading to a final retained austenite fraction of ~3 vol%. This sequence represents a typical secondary hardening response, analogous to that observed in carbon-containing tool steels [53,65,75].

The mechanical response after DA reflects the microstructural evolution induced by Mo₂C precipitation and the concurrent destabilization of retained austenite. The progressive depletion of Mo and C from intercellular austenite promotes its transformation during cooling, while nanoscale carbide precipitation increases resistance to dislocation

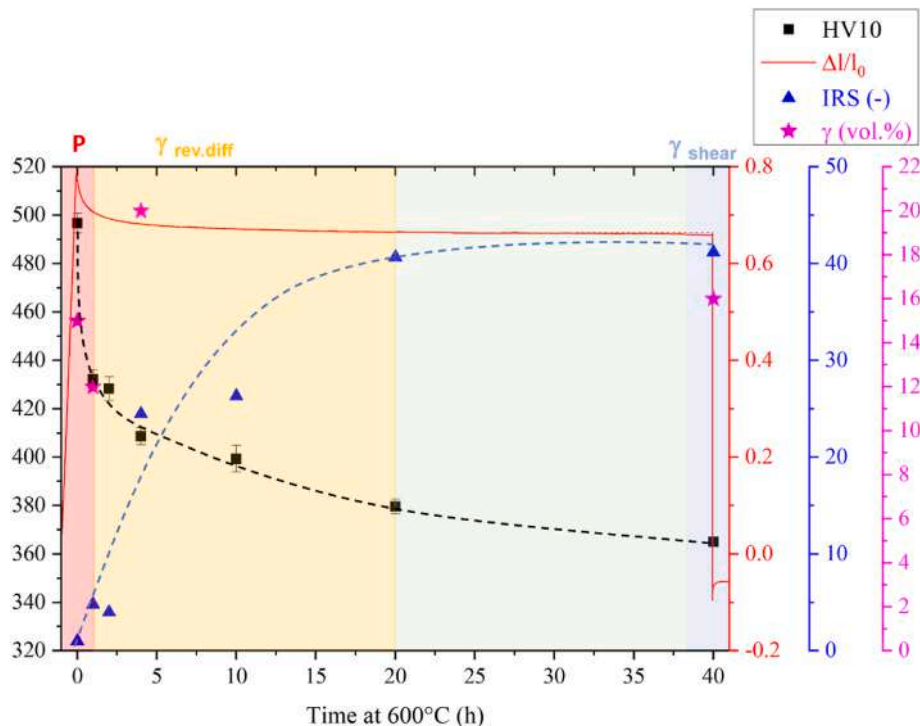


Fig. 18. The schematization of the phenomena occurring during tempering at 600°C for 40 h.

motion. As a consequence, ageing shifts the alloy from a mechanically heterogeneous microstructure, where deformation is partially accommodated by retained austenite, toward a carbide-strengthened martensitic structure characterized by higher resistance to plastic deformation. Accordingly, MAR55 exhibits a significant strengthening after DA, with increases of approximately 12% in yield strength and 17% in ultimate tensile strength compared to the AB condition, while retaining satisfactory ductility. This transition also explains the reduction in strength–ductility efficiency indicators observed after ageing, indicating that mechanical performance becomes increasingly governed by precipitation hardening rather than by the heterogeneous deformation mechanisms active in the AB condition. Nevertheless, MAR55 in the DA condition maintains a quite superior strength–ductility balance (21258 ± 964 MPa%) compared to 18Ni300 DA (20008 ± 408 MPa%), despite being comparable to 18Ni300 in the AB condition. Importantly, this performance is achieved at a substantially higher strength level (UTS ~ 1900 MPa for MAR55 DA versus ~ 1190 MPa for 18Ni300 AB), highlighting the retained synergy between strength and ductility in the MAR55 system after ageing. The tensile toughness of MAR55 DA (20026 ± 900 MPa%) remains comparable to that of 18Ni300 DA (19760 ± 411 MPa%), despite differences in absolute strength. Compared to 18Ni300, MAR55 exhibits significantly superior toughness, retaining nearly double the CVN impact energy after ageing (32 ± 3 J vs. 17 ± 1 J). This difference reflects the fundamentally distinct microstructural evolution and strengthening mechanisms developed during ageing. In MAR55, strengthening is governed by fine Mo_2C carbide precipitation within a predominantly martensitic matrix, whereas 18Ni300 derives its strength mainly from nanoscale $\text{Ni}_3(\text{Ti},\text{Mo})$ intermetallic precipitation accompanied by Ni-driven austenite reversion starting at approximately 480°C [22,23]. In the MAR55 AB condition, intercellular retained austenite contributes to deformation compatibility between adjacent regions, promoting more gradual damage accumulation under mechanical loading. Following DA, extensive carbide precipitation destabilizes the solute-enriched retained austenite, which partially transforms into martensite during cooling, progressively reducing the ability of the microstructure to accommodate localized stresses. Consequently, under dynamic loading conditions, crack nucleation occurs more readily, leading to the observed reduction in impact energy. A different behavior is observed in 18Ni300, where the significant drop in impact toughness after ageing (from ~ 108 J to ~ 17 J) is primarily driven by the intense precipitation of intermetallic $\text{Ni}_3(\text{Ti},\text{Mo})$ phases. While the presence of reverted austenite in 18Ni300 can provide a marginal toughening effect compared to a fully martensitic state, it also introduces locally softer regions that facilitate fatigue crack propagation through secondary cracking along intercellular boundaries. In contrast, the superior impact toughness of MAR55 DA (32 ± 3 J) is attributed to the transition to carbide-based strengthening (Mo_2C). These stable carbides ensure higher metallurgical compatibility and interfacial strength within the Fe–C martensitic matrix, effectively bridging the gap between high-strength tool steels and ductile maraging systems without triggering the severe embrittlement typical of traditional intermetallic systems at peak hardness [28]. The superior impact performance of MAR55 therefore suggests that a carbide-strengthened continuous martensitic matrix provides more effective resistance to crack propagation than the heterogeneous martensite–reverted austenite microstructure developed in aged 18Ni300.

Focusing on the thermal-conductivity behavior of MAR55 in comparison with 18Ni300 and wrought and AM H13 tool steels, the observed trends are primarily governed by alloy chemistry and microstructural evolution. High levels of Ni, Cr, Mo, and Si increase electron and phonon scattering, thus reducing thermal conductivity in heavily alloyed steels [76]. This explains the relatively lower thermal conductivity of 18Ni300 DA, where the high Ni content particularly limits heat transport. The marked increase in conductivity of MAR55 after direct ageing is attributed to carbide precipitation (depleting C, Mo, and Cr from solid solution) and to dislocation recovery, both of which enhance

electron and phonon transport. Interestingly, 18Ni300 exhibits an increase in thermal conductivity with temperature, reaching values similar to those of MAR55 and other steels around 500°C . This behavior can be explained by the combined effect of Ni-driven austenite reversion and microstructural recovery: the reduction in dislocation density and the transformation of reverted austenite, which has inherently higher conductivity than heavily dislocated martensite, facilitate heat transport at elevated temperatures. Wrought H13 - QT exhibits the highest conductivity across the investigated temperature range, due to its coarser, well-tempered martensitic microstructure, which reduces scattering interfaces and facilitates phonon transport. In contrast, additively manufactured H13 directly tempered (AM H13 DT) shows slightly lower conductivity despite similar ultimate tensile strengths (~ 1900 – 2000 MPa). This reduction is attributed to its finer cellular–dendritic microstructure, residual porosity, and elemental segregation typical of AM processing, which increases phonon scattering [76]. MAR55 DA achieves thermal conductivity comparable to AM H13 DT at low temperatures and surpasses it at higher temperatures, demonstrating the combined effects of carbide precipitation and dislocation recovery in enhancing heat transport. The combination of lower alloying content and effective microstructural stabilization in MAR55 allows for efficient thermal conduction, whereas 18Ni300 benefits from a temperature-dependent increase in conductivity due to Ni-driven austenite reversion and recovery, partially compensating for its higher alloy content. Nevertheless, even at elevated temperatures, MAR55 remains slightly below wrought H13 QT, confirming the superior thermal performance of conventionally processed tool steels.

Scratch tests provide direct insight into the wear resistance of the investigated alloys under repeated contact loading. The superior performance of MAR55 DA is attributed to its Fe–C martensitic matrix combined with stable Mo_2C carbides, which enhance resistance to plastic deformation and micro-cutting. In contrast, although 18Ni300 DA reaches higher hardness due to nanosized $\text{Ni}_3(\text{Ti},\text{Mo})$ precipitates, its softer Fe–Ni martensitic matrix and reverted austenite lead to a higher wear rate. These results indicate that scratch resistance is governed not only by bulk hardness, but also by the nature and stability of the strengthening phases, as confirmed by the narrower wear track observed for MAR55 DA (Supplementary File 2).

Overall, the mechanical performance of MAR55 is governed by the progressive evolution of its microstructure from a heterogeneous martensite–retained austenite architecture in the AB condition toward a precipitation-strengthened martensitic structure after DA. In the AB state, intercellular retained austenite promotes strain partitioning and sustained work hardening, enabling an exceptional combination of strength and ductility. After DA, precipitation of Mo_2C carbides increases strength and wear resistance by restricting dislocation mobility and improving resistance to localized surface deformation, although at the expense of reduced ductility and impact toughness due to the loss of retained austenite-mediated deformation mechanisms. These results demonstrate that controlling the balance between retained austenite stability and carbide precipitation is the key factor governing the overall mechanical behavior of MAR55.

5. Conclusion

This study provided a comprehensive assessment of the newly developed Osprey® MAR55 steel, focusing on its phase transformation behavior and the resulting mechanical and thermal properties in the as-built and directly aged conditions. The evolution of the material during heating was investigated to identify the main transformation stages and their role in the ageing response. The main findings are summarized as follows:

- L-PBFed MAR55 exhibits a predominantly martensitic microstructure containing ~ 15 vol% retained austenite, with a good combination of strength, hardness, toughness, and thermal conductivity in

the as-built condition. Compared with 18Ni300, MAR55 provides higher strength and hardness, although with lower impact toughness.

- Thermal analyses identified six transformation stages during heating to 900°C, from low-temperature carbon clustering and transition-carbide precipitation to retained-austenite decomposition, alloyed cementite and carbide precipitation, austenite reversion, and the final martensite-to-austenite transformation. Ageing at 480°C maximized carbide precipitation while limiting austenite reversion, resulting in improved microstructural stability and mechanical strength, whereas ageing at 600°C promoted austenite reversion and carbide coarsening, reducing hardness.
- Ageing at 480°C increased the hardness, yield strength, and UTS of MAR55 to ~550 HV10, ~1700 MPa, and ~1900 MPa, respectively, with an impact toughness of 32 J. Compared with 18Ni300, MAR55 showed lower hardness but higher toughness, higher thermal conductivity over the investigated temperature range, and promising wear resistance despite its lower hardness. Its thermal conductivity was also comparable to that of AM H13 at low temperatures and higher at elevated temperatures.

Overall, MAR55 demonstrates an optimized balance of mechanical and thermal properties, effectively bridging the gap between Fe–Ni maraging steels and Fe–C hot-work tool steels. Its microstructural stability, precipitation hardening response, and heat transport capabilities make it particularly suitable for high-performance AM applications where both mechanical robustness and thermal resistance are required.

CRediT authorship contribution statement

Lorena Emanuelli: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Ville-Pekka Matilainen:** Writing – review & editing, Investigation. **Francesca Russo:** Methodology, Investigation. **Lorena Maines:** Methodology, Investigation. **Jonathan Hann:** Methodology, Investigation. **Arne Röttger:** Writing – review & editing, Writing – original draft, Supervision. **Massimo Pellizzari:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation. **Faraz Deirmina:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Methodology, Investigation, Conceptualization.

Declaration of generative AI use

During the preparation of this work the authors used ChatGPT (based on GPT-4 - OpenAI) in order to reorganize the notes and improve the manuscript readability. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

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Declaration of competing interest

Co-author Faraz Deirmina was an employee of Sandvik Additive Manufacturing. This study was conducted independently, and Sandvik did not provide direct funding or influence the design, analysis, or interpretation of the research. The authors have studied a product from Sandvik as part of this research. All decisions regarding study design, data collection, analysis, and publication were made independently of Sandvik.

No other financial or non-financial competing interests exist.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.msea.2026.151066>.

Data availability

Data will be made available on request.

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