Thermal aging of additively manufactured maraging steel

Micaela Davids[1,3*], *Charmaine* Johnston[1], and *Lerato* Tshabalala[2]

[1] Defence and Security, Council for Scientific Industrial Research, South Africa
[2] Future Production: Manufacturing, Council for Scientific Industrial Research, South Africa
[3] School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand, South Africa

Abstract. The mechanical properties of Additively Manufactured M300 Maraging Steel were tested after two thermal treatments. It was found that the stress-relief process resulted in tensile strength comparable to the powder specification, but low ductility compared to the specifications, whilst the addition of an aging process resulted in substandard tensile strength and extreme brittleness. The results of a hardness test and the tensile properties achieved suggest that the material was embrittled by both thermal processes. The samples were inspected using an Optical Microscope (OM) after testing.

1 Introduction

Historically, thermal treatment of metals has been used as a method of improving their mechanical properties, specifically the tensile strength and hardness [1]. M300 Maraging Steel is stress-relieved by the material manufacturer at high temperatures, to aid in eliminating residual stresses. It may then be subjected to another thermal treatment such as aging during component manufacturing, to further improve the mechanical properties. [2]. M300 is used for manufacture because it is easy to machine [2].

Ideally that the M300 metals produced using Selective Laser Melting (SLM) techniques have similar properties to the wrought M300 metals [3]; this should be true for the metal in the aged condition as well. According to most M300 powder datasheets, the properties will vary slightly depending on print orientation but will be comparable to conventionally manufactured (wrought) M300 [1, 4]. The characteristics and performance of AM metals may be affected by defects arising from the AM technique used, the machine used, or the build parameters used. These defects can include porosity, density variations, surface defects and/or residual stresses [5]. These defects can be mitigated with heat treatments and appropriate build processes.

The generally accepted methods of thermal treatments relevant to this research are as follows:

Solution annealing – The material is heated to 815°C for 1 hour, then air cooled. This is done to relieve residual stresses. Solution annealing is usually done by manufacturers

* Corresponding author: 2103735@students.wits.ac.za

before distribution if the material is needed for machining applications. A higher strength can be achieved with shorter annealing times [1].

Age hardening - After solution annealing, the alloy is heated to within 455-510°C, held for 3-12 hours, and then air cooled to room temperature. It has also been shown that long holding times can increase tensile strength significantly, but this is predicted to be in the order of hundreds of hours. The percentage of elongation is not typically expected to drop below 10%, no matter how long the holding time [1].

Over-aging – The material is heated in the range of 500-650°C for 6 hours, then air cooled. The exact relationship between temperature, time and mechanical properties can be complex. The best Ultimate Tensile Stress (UTS) and Yield has generally been achieved after aging at 550°C. The best percentage of elongation is generally achieved after aging at 650°C. Percentage of elongation should increase with temperature and time. [1, 6].

It is expected that, within these temperature ranges, the M300 will remain in the ferrite, and austenite-ferrite stages [7].

The objectives of this research are to determine the mechanical properties of AM M300 Maraging steel after stress-relieving, and after stress-relieving and aging, at high temperatures and extended holding time. It will then be established whether a combination of thermal treatments is beneficial for the properties of this AM steel.

2 Methodology

2.1 Samples

Five samples were printed according to the ASTM E8 standard for tensile testing [8,9]. They were printed in an EOS M280 printer, at a 90° orientation, with bidirectional laser movement. The laser was also rotated by 47° between each layer. The powder size used was approximately 20-60 microns in diameter. The geometry of the samples is given in Fig. 1, and the samples were printed in Z-direction.

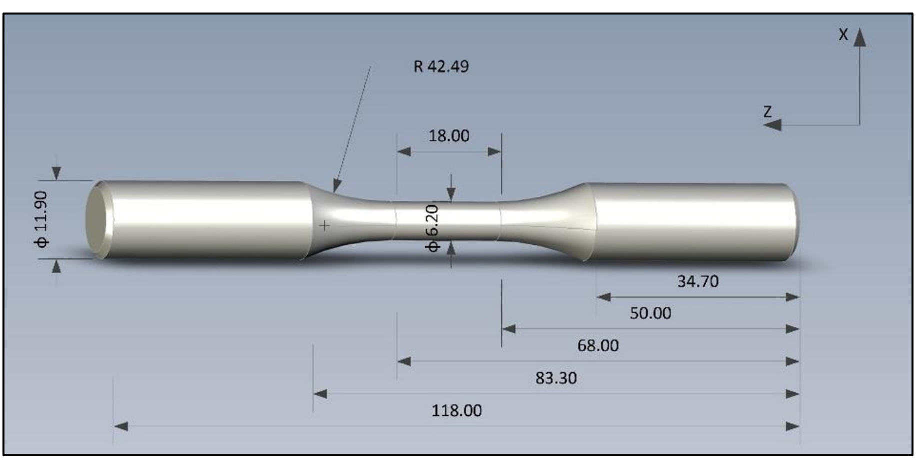

Fig. 1: Dimensions of the test specimen

The samples were Stress-Relieved (SR) at 890°C for 3 hours and furnace-cooled, according to the manufacturer's standard procedure. The samples underwent threading (M12x1) at the grip sections and were then turned along the length to smooth the surface (see Fig. 2). Finally, three of the samples were aged at 480°C for 3 hours (Stress-Relieved and Aged, SRA) and then air cooled.

Fig. 2. Samples after Stress-relieving and Aging processes

Table 1 The chemical composition (in weight percentage) of three samples was determined for consistency, using Spark-Optical Emission Spectrometry (OES). This method uses sparks to cause light emission from the material. The wavelengths produced are then analysed to determine the elements present in the composition [10].

2.2 Mechanical properties

All five samples underwent tensile testing in the same machine, an Instron-1342, which has a maximum loading capacity of 30kN.The tests were done according to the ASTM E-8M standard. Up to 2% strain (the yield strength), the strain rate is 0.5mm/min. The extensometer is then removed, and the strain rate is increased to 1.5mm/min until failure.

The samples were screwed into the grips by the threaded sections on each end and were then clamped securely to avoid slippage. Tensile testing requires complete breakage of the samples, as they are loaded until failure. This means that each test can only be done once for each sample, but an average can be obtained for multiple samples. The results of the tensile tests were recorded, and an average was obtained for the stress-relieved samples and the aged samples. The results of interest were the percentage of elongation and the ultimate tensile strength of the samples. The samples were also tested for hardness. The strain in each sample was measured using strain gauge data, with the strain gauges connected directly to the test sections of the samples. The relationships between the force applied by the machine and the strain measured from the sample could then be used to calculate the Yield Stress, UTS, percentage elongation, and Young's Modulus.

The cross-sectional area (mm^2) of each sample was calculated from the measurement of the test-section diameter. These measurements were taken using a set of vernier callipers. The Yield and UTS (in MPa) could then be calculated using the force that was applied at the point of plastic deformation and of total failure of the sample, respectively. The hardness (HV) was measured using a Zwick-Roell testing machine, with a total of 21 indentations at a force of 300g, taken at the grip section of two samples (one SR and one SRA).

All tests were conducted in a controlled environment at 20°C to ensure consistency.

2.3 Optical Micrographs

The samples were subsequently viewed under an Optical Microscope (OM) to determine the state of the microstructure and grain orientations. The OM used was a Leica DMI5000 M.

The samples were etched using modified Fry's reagent (40% HCl, 5% $CuCl_2$, 25% C_2H_5OH, Bal H_2O) [11].

3 Results

3.1 Samples

The chemical composition (in weight percentage) of two samples was determined for consistency, with results shown in Table 1.

Table 1. Chemical composition of M300 maraging steel [% by weight]

Metal	C	Ni	Co	Mo	Ti	Al	Fe
M300 Standard [1]	0.03 max	17.0 -19.0	8.5 - 9.5	4.5 - 5.2	0.6 - 0.8	0.05-0.15	Bal
AM Sample (Ave)	0.011 ±0.0005	17.105 ±0.015	9.515 ±0.015	5.140 ±0.06	1.625 ±0.065	0.135 ±0.003	Bal

While the Titanium content is markedly higher in the AM samples, this is not expected to have a significant impact on the formation of precipitates during the heat treatment processes. This is because the Carbon content is negligible, and the formation of TiC precipitates requires a high Carbon content [12]. This is important to consider as TiC precipitates can cause a material to be brittle, especially when undergoing heat-treatments.[1]

3.2 Mechanical properties

The results of the tensile testing are summarised in Table 2 and Fig. 3 for the SR samples, and in Table 3 and Fig. 4 for the SRA samples. The properties according to the manufacturer datasheet for Additively Manufactured TruForm M300 Metal Powder is titled "Powder spec.". The properties for wrought M300 are also shown in the tables.

Some samples failed without first reaching a measurable yield strength (i.e. there was no elastic deformation), and so the values for a 0.2% yield are not all reflected in the tables.

Table 2. Mechanical properties of the SR samples

	Powder Spec. [4]	**SR1**	**SR2**	**Average SR**	**Wrought M300** [13]
Yield [MPa]	990	916.67	877.82	897.2 ± 19.42	790
UTS [MPa]	1 100	1 192.41	1 192.44	1 192 ± 0.01	1 010
Elongation [%]	15	8.04	6.54	7.29 ± 0.75	17
Hardness [HV]	340	-	383.281	-	320

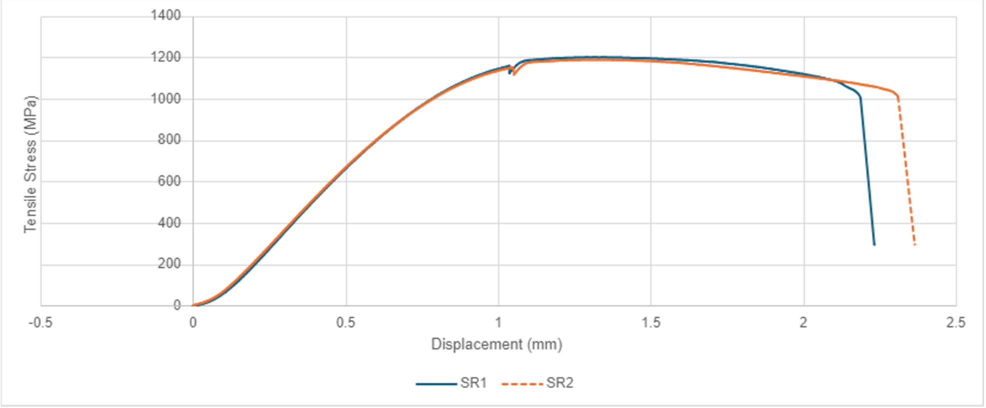

Fig. 3. Stress/strain characteristics for the Stress Relieved (SR) samples

The results from the SR samples were comparable to the tensile strength of wrought M300, however, the stress-relieving process seems to have compromised the ductility of the material. Ideally, laser-fused AM Maraging Steels should achieve an elongation value of approximately 14.4% after solution annealing [14]. The 7.29% obtained suggests that the embrittlement process has already begun in the first thermal treatment process. Moreover, the hardness of the AM SR samples is almost 20% higher than what the wrought M300 is, indicating that the thermal treatment process has over-hardened the material. This unexpected data could be due to the temperature of the treatment being high compared to the standard for M300 (890°C, as opposed to 815°C) [1].

Table 3. Mechanical properties of the SRA samples

	Powder Spec. [4]	**SRA1**	**SRA2**	**SRA3**	**Average SRA**	**Wrought M300** [13]
Yield [MPa]	2 100	-	1 025.64	-	-	1 930
UTS [MPa]	2 105	1 708.71	1 488.84	1 497.18	1 565 ± 101.74	1 965
Elongation [%]	6	0.13	0.46	0.17	0.25 ± 0.15	7.5
Hardness [HV]	610	-	683.33	-	-	560

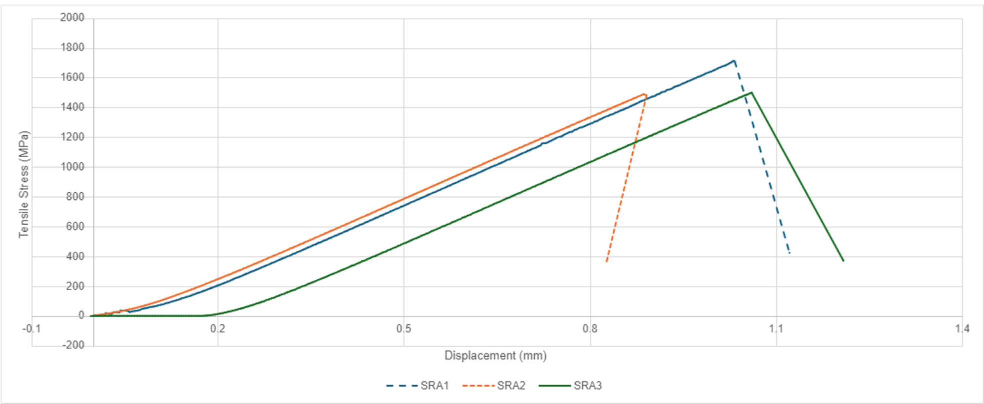

Fig. 4. Stress/strain characteristics for the Stress Relieved and Aged (SRA) samples

The SRA samples performed poorly compared to wrought M300 in all areas except hardness. The drastic reduction in ductility, as well as the 21% increase in hardness of the SRA samples indicated that the samples had been significantly embrittled by the heat treatment processes. It may be possible, however, that the air-cooling process contributed to the high hardness achieved, as fast-cooling (as opposed to slower, furnace-cooling) is expected to generate higher hardness in steels [15].

It is expected that laser-fused AM Maraging Steels achieve approximately 3.6% elongation after aging [14]. The samples achieved 0.25% elongation and are clearly brittle and overaged. The hardness of 683.33HV measured exceeds that found in a double-aging experiment performed by Pérez-Gonzalo *et al.* [12], where the maximum hardness did not exceed 610HV.

3.3 Optical micrographs

The microstructures of the samples, as seen using the Optical Micrograph (OM) technique, are presented in Fig. 6 (stress relieved) and Fig. 7 (stress relieved and aged), for comparison to stress-relieved wrought M300 metal from the work of Chakravarthi *et al* [16] (Fig. 5).

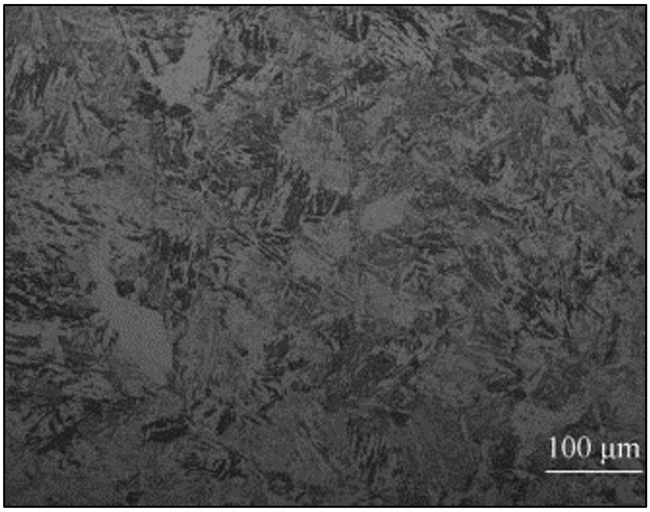

Fig. 5. OM of stress-relieved wrought M300 metal at 100 micron [16]

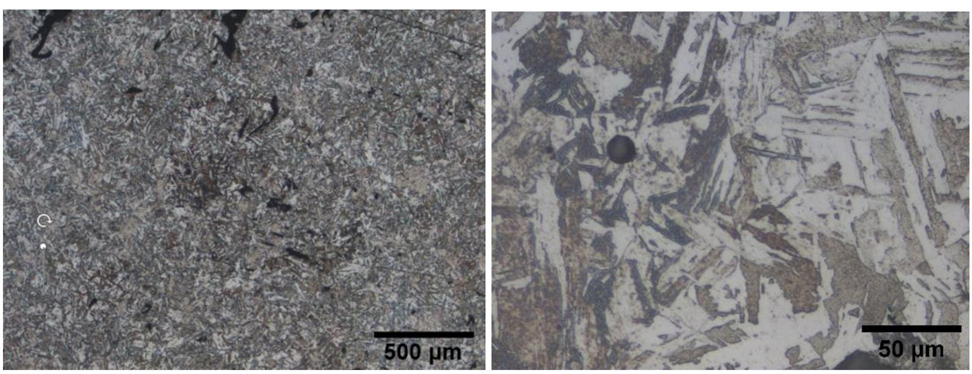

Fig. 6. OM of stress-relieved (SR) AM M300, at lower and higher magnification, from SR2

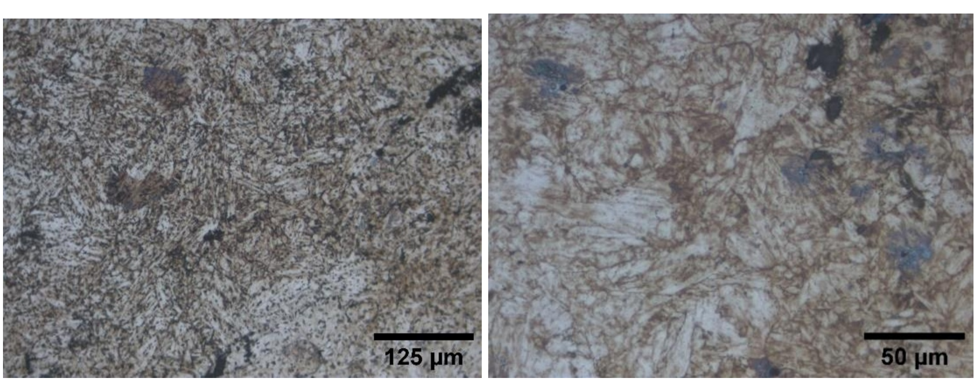

Fig. 7. OM of stress-relieved and aged (SRA) AM M300 at lower and higher magnification, from SRA2

The microstructure of the SR sample in Fig. 6 shows lath martensitic structure in the annealed condition, with slightly refined grains from that of the M300 microstructure in literature, see Fig. 5. The Hall-Petch relation states that a smaller grain size relates to an increase in tensile strength [17], and this is evident in the tensile test results where the AM SR samples performed better than the wrought M300 steel (Fig. 3). In the higher magnification scans (right) for both samples, the images show differences in the grain sizes and orientations.

The SR sample has a much coarser grain structure, whilst the SRA sample has a more refined finer grain structure, see Fig 7. Thermal aging (SRA) increased the tensile strength but not to value in the specification (Fig. 4).

On the lower magnification scans (left) of the SR and SRA samples, some melt-pools and evidence of keyhole porosity can be seen. Usually this occurs when the movement and heat of the laser cause the melted powder to collect and form a cavity. Keyhole porosity is a result of diffused gas from the powder particles becoming trapped in the material [14]. These defects can give rise to residual stresses in the structure, making it more prone to cracking when introduced to higher temperatures (such as in the case of a heat treatment) [14]. The presence of pearlite may be seen in both micrographs, but no evidence of austenitic grain boundaries, thus the samples remained within the ferrite phase before and after the heat treatment processes [7].

4 Conclusion and recommendations

It is evident from the results obtained that the dual thermal treatments used in this study caused embrittlement and reduced strength in AM M300, in which condition the material does not meet the published specifications. This effect appears to be due to grain refinement of heterogeneous structures.

The stress-relief and aging processes will be investigated further in future. Computational Topography (CT) techniques and OM analysis may also be used to further understand the internal structures that have direct impact on the mechanical properties. It may also be beneficial to analyse the samples using a Scanning Electron Microscope (SEM) between each heat treatment to visualise the changes with respect to a phase transformation diagram for this metal.

The authors wish to acknowledge and thank the South African Air Force (SAAF) and the Armaments Corporation of South Africa (ARMSCOR) for their support of this project.

References

1 *ASM Handbook* ***Volume 4D*** *Heat Treating of Irons and Steels*, (1991)
2 A.P. Mouritz, *Introduction to Aerospace Materials,* ***Chapter 11****: Steels for aircraft structures*, pages 232–250, Woodhead Publishing, (2012)
3 E. Yasa, K. Kempen, J.-P. Kruth, L. Thijs, J. van Humbeeck, *Microstructure and Mechanical Properties of Maraging Steel 300 after Selective Laser Melting*, page 383
4 AM Quality Lab Powder Atomization Capabilities TruFormTM Metal Powders for Additive Manufacturing (2022) [Online]. Available: https://www.i3dmfg.com/wp-content/uploads/2019/01/TruForm-Maraging-Steel.pdf
5 A. Mostafaei *et al.*, *Defects and anomalies in powder bed fusion metal additive manufacturing*, Current Opinion in Solid State and Materials Science, 26 (2022)
6 F. Tariq, M. Shifa, R.A. Baloch, *Effect of Overaging Conditions on Microstructure and Mechanical Properties of Maraging Steel*, Metal Science and Heat Treatment, **Vol. 62**, pages 188–194 (2020)

7 D.S. Mackenzie, *Back to basics on heat-treatment of steel, underlying metallurgy* (2018) [Online]: https://gearsolutions.com/media/uploads/2018/09/0918-HS.pdf
8 ASTM International, 2018 Annual Book of ASTM Standards **Volume 03.01** (2018)
9 C.M. Johnston, L.C. Tshabalala, M. Davids, *Fatigue properties of additively manufactured tool steel*, 34th Congress of the international council for the aeronautical sciences (ICAS), Florence, Italy, Sep 2024, presented on the 11th of September 2024
10 ThermoFisher Scientific. (n.d.). Spark-Optical Emission Spectrometry (OES). *ThermoFisher Scientific*. Retrieved October 1, 2024, from https://www.thermofisher.com/za/en/home/global/forms/industrial/oes-periodic-table-quick-guide-download.html
11 Australian Chemical Reagents, Safety Data Sheet (2022)
12 I. Pérez-Gonzalo, A. González-Pociño, F. Alvarez-Antolin, and L. del Rio-Fernández, *Analysis of a Double Aging Process in a Maraging 300 Steel Fabricated by Selective Laser Melting, Using the Design of Experiments Technique*, Metals, 13 (2023)
13 18% Nickel Maraging Steel-engineering Properties: A Practical Guide to the Use of Nickel-containing Alloys NO4419 [Online]. Available: www.nickelinstitute.org
14 S. Ramadurga Narasimharaju *et al.*, *A comprehensive review on laser powder bed fusion of steels: Processing, microstructure, defects and control methods, mechanical properties, current challenges and future trends*, in Jour of Manuf Process 75 (2022)
15 M. Król, P. Snopiński, J. Hajnyš, M. Pagáč, and D. Łukowiec, *Selective Laser Melting of 18NI-300 Maraging Steel*, Materials (2020)
16 K.V.A. Chakravarthi, N.T.B.N. Koundinya, S.V.S. Narayana Murty, B.N. Rao, Microstructure, properties and hot workability of M300 grade maraging steel (2017)
17 N. Hansen, *Hall-Petch relation and boundary strengthening*, Scripta Materialia Volume 51 pages 801–806 (2004)