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Research article

## On the effect of pre-strain and pre-fatigue on the monotonic behaviour of ultra-high strength steels



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#### ABSTRACT

The mechanical behaviour of three ultra-high strength steels has been assessed; AerMet100, 300M and the recently developed corrosion resistant high strength steel, MLX-17. Material heat treatment profiles have been utilised to provide performance optimised for the aerospace industry and specimens have been tested to explore tensile and fatigue properties, in particular when combined with pre-strain to simulate the effects of overload. Testing of this kind has not been reported within the literature, particularly amongst ultra-high strength and corrosion-resistant steels. Baseline mechanical performance for all three materials in their heat-treated conditions has been established and properties such as yield strength and ultimate tensile strength have been assessed following a 75% and 95% pre-strain as well as fatigue in combination with a 75% and 95% pre-strain. Under all loading conditions, resultant tensile mechanical properties are not seen to witness a substantial degradation in performance, but an improvement in terms of yield strength and UTS, due to the role of work hardening. An alloy comparison has been carried out and responses are seen to vary slightly as a result of material microstructure. Correlation of pre-strain and pre-fatigue results with respect to baseline properties and microstructure has contributed to advancing the understanding of the mechanical behaviour of the aforementioned ultra-high strength steels.

#### 1. Introduction

Ultra-high strength steels (UHSS) are a classification of structural steels that are typically utilised at minimum yield stresses of 1380 MPa [1]. Their high yield coupled with high ultimate tensile strength (UTS), reasonable ductility and superior fracture toughness, makes this classification of steels suitable for aerospace applications, in particular for use in landing gear components [2]. AerMet100 and 300M are both currently widely used, martensitic UHSS's that require a cadmium coating to provide their corrosion resistance. Due to EU legislation, however, the use of cadmium plating for general engineering purposes is no longer permitted as a result of its carcinogenic attributes and the high toxicity of the application process [3]. Airframe manufacturers, are therefore being compelled to consider alternative materials which offer improved corrosion resistance as an inherent property, combined with similar mechanical properties offered by existing alloys. In response to industry requirements, corrosion resistant high strength steels (CRHSS), such as the newly developed MLX17 have been introduced [4]. In order to meet high levels of corrosion resistance, these novel alloys have substantial Cr additions (>11.5%wt) when compared to their predecessors but maintain superior strength and fracture toughness properties, which is primarily owed to their martensitic structure.

Lee *et al* [5] have previously conducted various tensile and fatigue tests on a range of aerospace steels including 300M and AerMet100 in order to characterise and compare their mechanical properties. Results show that 300M exhibits a  $K_{IC}$  of 57 MPa $\sqrt{m}$  and a UTS of  $\sim$ 2000 MPa and AerMet100, a  $K_{IC}$  of 125 MPa $\sqrt{m}$  and UTS of 1979MPa. MLX17 is reported to have slightly lower UTS values of up to  $\sim$ 1700 MPa, depending on heat treatment [4].

During service, landing gear are inherently subjected to a variety of substantial loading conditions, including low cycle fatigue, high cycle fatigue and overloads due to heavy landings. Failure of such components is commonly attributed to fatigue, corrosion, shear, bending and tensile stresses, including overload [6, 7, 8] The magnitude of these loads is influenced by a number of different factors, such as weather, descent velocity and human error, resulting unpredictable loading profiles. As such, it is essential that aircraft and alloy manufacturers ensure that materials can withstand such conditions. Although an understanding of

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the baseline mechanical properties of these materials is established, there is an apparent lack of research exploring alloy response to representative complex loading conditions. Therefore, in order to address this, the effects of varying magnitudes of pre-loading and pre-fatigue on the monotonic mechanical properties will be investigated, which will be essential in determining the suitability of these alloys for landing gear applications. In addition, it is important to gain a comprehensive understanding of all alloys baseline tensile behaviour and to characterise and compare the mechanical properties of existing UHSS to newly developed, novel CRHSS. This paper therefore aims to assess and compare the effect of various loading conditions (pre-strain, overload and pre-fatigue) on the monotonic performance of; AerMet100, 300M and MLX17.

#### 2. Materials and methods

#### 2.1. Ultra-high strength and corrosion resistant steels

Three steels were considered for this research paper; ultra-high strength steels AerMet100 and 300M, and the more recently developed, corrosion-resistant high strength steel MLX17. Such examples typically witness an improved fracture toughness through the addition or control of non-metallic inclusions [9]. All alloys were received in their normalised condition and heat treated as recommended by the manufacturers to achieve the desired properties for landing gear application. Once heat treated, alloys were mounted in Bakelite, ground and polished to a final stage of OPS Colloidal Silica in preparation for etching. Etching was performed utilising the methods and solutions shown in Table 1.

Latrobe Lescalloy® 300M steel is a medium carbon, modified 4340 steel with added silicon allowing for use at high temperatures. Its most commonly used in its quenched and tempered martensitic condition where it is at its highest strength and toughness. The addition of silicon also acts to retard the coarsening of the cementite phase, allowing the 300M to be tempered at much lower temperatures, thus avoiding the loss of strength [10]. Its composition is shown in Table 2.

300M undergoes VIM-VAR processing, providing it with higher strength than 4340, but similar fracture toughness. As recommended, 300M underwent the following heat treatment: 927 °C for 1 h  $\rightarrow$  870 °C for 1 h  $\rightarrow$  oil quench to room temperature  $\rightarrow$  double temper at 300 °C for 2 h each  $\rightarrow$  air cool to room temperature. The heat treatment resulted in the martensitic structure as shown in Figure 1.

CarTech's® AerMet100 is considered a high alloy steel due to it containing large amounts of Cr, Ni and Co. It is a martensitic steel with an Fe–Ni lath structure which is strengthened by  $M_2C$  (M=Cr, M0 and Fe) carbides [11]. Its chemical composition is shown in Table 3.

It receives a similar VIM-VAR process as 300M, which gives it a clean, inclusion-free matrix. AerMet100 also receives a cryogenic treatment after treatment after quenching to room temperature following austenitising. This is to ensure the majority of the retained austenite is transformed to martensite. The addition of Co increases  $M_2C$  carbide nucleation as well as increasing particle reinforcement [12]. Heat

treatment of AerMet100 was carried out as follows: 899 °C for 1 h  $\rightarrow$  air cool to room temperature  $\rightarrow$  885 °C for 1 h  $\rightarrow$  quench to room temperature over 1–2 h  $\rightarrow$  refrigerate at -73 °C for 1 h  $\rightarrow$  air heat to room temperature  $\rightarrow$  temper at 482 °C for 5 h  $\rightarrow$  air cool to room temperature. This resulted in a fully martensitic structure as shown in Figure 2.

MLX17 is a corrosion resistant high strength steel developed by Aubert & Duval. Its high corrosion resistance is attributed to its comparably high %weight of Cr and Ni and it is precipitation hardened where Al and Ti are hardening additions [4]. The chemical composition of MLX17 is shown in Table 4.

MLX17 is a high purity alloy, manufactured through vacuum primary melting and consumable electrode remelting. It was received in its cryogenic solution treated condition, however, to achieve optimum hardness values, ageing was carried out at 510  $^{\circ}$ C for 7 h followed by an air cool at room temperature. The heat treatment resulted in a martensitic structure as shown in Figure 3.

Since MLX17 is a more recently developed alloy, little research has been conducted into its baseline mechanical properties, hence a key aim of this paper is to further the current understanding of its monotonic behaviour under various loading conditions whilst comparing its properties to existing alloys.

#### 2.2. Test specimens

For all monotonic and fatigue testing, round-bar specimens were utilised with a diameter of 4.5mm and a gauge length of 12mm with a tolerance of +/- 0.05mm. Subsequent to material specific heat treatments, blanks were machined into mechanical test specimens and ground to the final profile with a surface finish of 0.25mm. For all test types presented within this paper, tensile and fatigue, three repeat tests were conducted to ensure consistency of baseline values and results generated under complex loading. Data presented and plotted shows an average value of three repeat tests.

#### 2.3. Baseline monotonic testing

Prior to exploring the effects of pre-loading and pre-fatigue, it was essential to obtain the baseline monotonic mechanical properties of each material. An average value for UTS is then obtained from three repeat tests and used to determine required loads for pre-straining the specimens at 75% and 95%. Other vital properties such as yield strength, Youngs Modulus, 0.2% proof stress and elongation can also be determined from these tests. All tensile tests were performed on a 50kN capacity, universal servo-hydraulic test frame at room temperature (21  $^{\circ}$ C), utilising a strain rate of 0.12 mm min $^{-1}$  and monitored with a 12mm MTS extensometer.

#### 2.4. 75% and 95% pre-strain testing

In order to simulate the effect of overload due to heavy landing, specimens were pre-strained to 75% and 95% of the materials' UTS

Table 1. Etching procedure for all steel alloys assessed.

Material	Etchant	Method	Application Time
300M	5% Nital	Submerge	4–5 s
AerMet100	5% Nital	Submerge	4–5 s
MLX17	Kallings Reagent 1	Submerge	1–3 s

Table 2. Chemical composition of 300M (%wt).

С	Cr	Si	V	Mn	Al	Sn	P	S	Ti	Ni	Fe
0.43	0.35	1.3	0.1	0.9	0.18	0.02	0.01	0.008	0.01	2.0	Bal

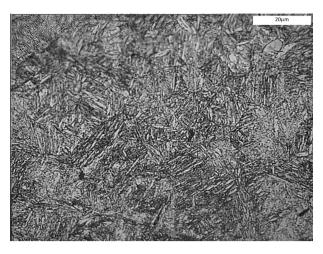


Figure 1. Optical micrograph of 300M microstructure.

before undergoing a tensile pull to failure. Loads for pre-straining were calculated utilising values of UTS obtained from baseline monotonic tests and are shown in Table 5. Using Dirlik Controls tensile test software, specimens were loaded to the pre-strain value at a strain rate of 0.12 mm  $\rm min^{-1}$ . Once the pre-straining load was achieved, the test was stopped, and load removed. The specimen was then pulled under tension at a similar strain rate until complete failure. Again, three repeats were carried out for each test and data was averaged.

#### 2.5. Fatigue testing +75% and 95% overload

To assess the influence of pre-fatigue combined with static overload on the materials' mechanical properties, specimens were subjected to a 1Hz sinusoidal fatigue waveform, with an R ratio of 0.1, for 25% of the predicted in-service fatigue life (3000 cycles). A peak stress of 1200 MPa was selected to ensure that fatigue damage was induced in the samples prior to overloading, whilst also similarly representing a mid-range inservice stress amplitude. Testing was carried out on a servo-hydraulic test frame under load control. Once specimens reached 3000 fatigue cycles, an overload of either 75% or 95% of the UTS was applied using similar strain rates as mentioned previously, followed by a return to zero load and a subsequent tensile pull to failure.

#### 3. Results and discussion

#### 3.1. Summary of results

Table 6 provides a summary of key numeric values, including UTS, yield strength, 0.2% proof stress, Youngs Modulus and elongation for all data generated, as described above. When analysing data, the yield strength is considered the point at which the linear elastic region deviates from the proportionality limit. The 0.2% proof stress is then calculated by generating a curve of equal gradient to that of the linear elastic region,

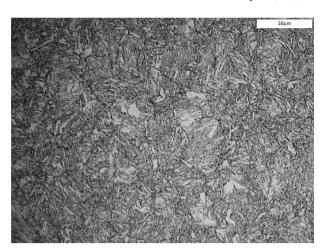


Figure 2. Optical micrograph of AerMet100 microstructure.

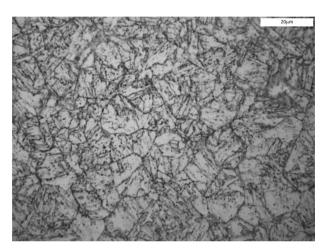


Figure 3. Optical micrograph of MLX17 microstructure.

offsetting the curve by 0.2% and taking the point at which this intercepts with the stress-strain curve generated.

#### 3.2. Baseline monotonic testing

Using average values obtained during tensile testing, plots of stress vs strain were constructed. Figure 4, along with Table 6 show the baseline mechanical properties for 300M, AerMet100 and MLX17 following their respective heat treatments. Little scatter was observed amongst tests and values for UTS and yield were consistent. It is confirmed that the current classification of HSS's have both higher UTS and yield stress values than their newly developed counterpart. 300M has the highest UTS value of 2031 MPa, followed by AerMet 100 with 1996 MPa and MLX17 with 1722 MPa.

Table 3. Chemica	composition	of AerMet100 (%wt).
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С	Ni	Со	Cr	Мо	Fe
0.23	11.1	13.4	3.1	1.2	Bal

Table 4. Chemical composition of MLX17 (%wt).

С	Si	Mn	Cr	Mo	Ni	Al	Ti	Fe
< 0.2	< 0.25	< 0.25	12	2	11	1.5	0.3	Bal

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% pre-strain	Material					
	300M	AerMet100	MLX17			
75%	1523 MPa	1478 MPa	1291 MPa			
95%	1927 MPa	1864 MPa	1607 MPa			

Table 6. Summary of all data generated for all materials.

		UTS (MPa)	Yield (MPa)	0.2% Proof (MPa)	Youngs Modulus (GPa)	Elongation (%)
300M	Tensile	1360	1582	1693	207	15.3
	75% PS	1421	1646	1728	220	15
	95% PS	1722	1964	1980	187	15.3
	Fatigue +75% OL	1565	1660	1734	194	15.8
	Fatigue +95% OL	1748	1842	1894	188	16.1
AerMet 100	Tensile	1996	1456	1762	184	22.3
	75% PS	1998	1544	1772	197	21.4
	95% PS	1945	1744	1879	177	21.4
	Fatigue +75% OL	2009	1569	1797	192	22.6
	Fatigue +95% OL	1919	1708	1868	175	22.1
MLX 17	Tensile	1722	1373	1650	203	19.4
	75% PS	1788	1550	1718	187	19.4
	95% PS	1774	1667	1761	190	19.7
	Fatigue +75% OL	1823	1556	1757	198	19
	Fatigue +95% OL	1758	1642	1718	188	19.4

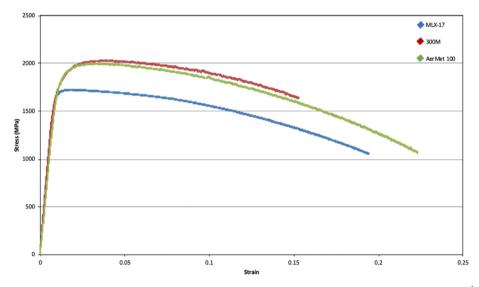


Figure 4. Comparison of baseline engineering stress vs engineering strain for all materials, conducted at 0.12 mm min<sup>-1</sup> strain rate.

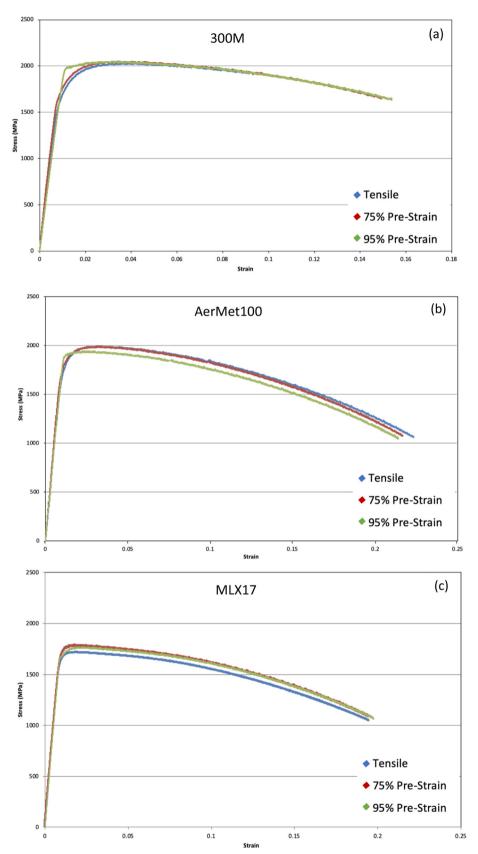
#### 3.3. 75% and 95% pre-strain testing

In order to simulate a near-UTS overload event, specimens were prestrained to 75% and 95%, followed by testing to failure. Figure 5a–c illustrate the tensile curves following pre-straining for all three materials.

It's clear that pre-straining has slightly different effects on each material, however the majority of materials demonstrate changes in both UTS and yield stress when applied with pre-strain. Results show that with increasing pre-strain, 300M, AerMet100 and MLX17 all undergo a slight increase in UTS, with 300M peaking at 95% pre-strain and AerMet100 and MLX17 showing a maximum UTS at 75% pre-strain. Similar observations can also be made for yield strength and 0.2% proof stress, where all materials demonstrate increasing values with increasing pre-strain. Values for Youngs Modulus, on the other hand can be seen to peak at

75% pre-strain for current UHSS's, but the opposite is witnessed in the new CRHSS, MLX17. In terms of elongation, minimal effects are seen throughout all three materials.

The increase in yield strength is the most significant modification, particularly when specimens were exposed to a 95% pre-strain, where an increase of up to 400MPa was observed. This effect of increasing yield with increasing pre-strain is expected and discussed in detail in the literature [13, 14]. Peng *et al* illustrates that with increasing pre-strain, a corresponding increase in the value of yield stress is often observed [15]. It has been well established that the increase in yield is due to strain induced mechanical twinning and the multiplication of dislocations, with an increase in relative dislocation density being observed with increasing pre-strain [16, 17]. High dislocation density often results in phenomena such as stacking, dislocation pile-up and pinning, all hindering



 $\textbf{Figure 5.} \ \ \text{Baseline tensile, } 75\% \ \ \text{and } 95\% \ \ \text{pre-strain stress-strain curves for a) } 300M, \ \ \text{b)} \ \ \text{AerMet100, c)} \ \ \text{MLX17.}$ 

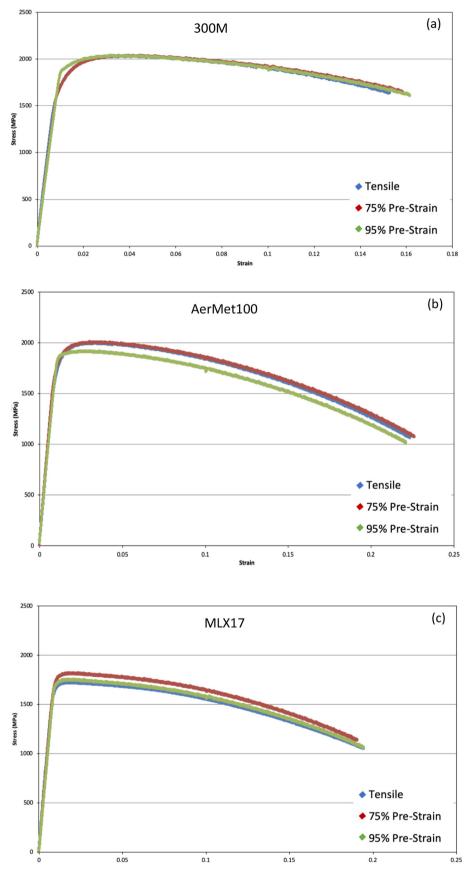


Figure 6. Stress-strain data following pre-fatigue and overloads of 75% and 95% for a) 300M, b) AerMet100, c) MLX17.

dislocation movement and thus strengthening the material. When failure is desired, a higher applied stress is therefore required to remobilise dislocations, therefore resulting in a higher magnitude of yield strength.

The most notable increase in yield is observed in 300M, Figure 5a, where a low yield to UTS ratio is observed following a 95% pre-strain, illustrated by a peak in the curve. Unlike AerMet 100 and MLX17, 300M does not undergo a cryogenic heat treatment to achieve a fully martensitic structure and is therefore likely to experience some levels of retained austenite. It has been shown in previous studies, for austenitic steels, that at high levels of strain, an increase in the density of martensite transformation occurs [18]. It is possible that in the case of 300M, when undergoing 95% pre-strain, the retained austenite in the structure undergoes transformation to the strengthening martensite phase, which, when tested to failure, results in higher yield stress values.

It is important to note, that even upon pre-straining up to 95% of UTS in all four materials, no detrimental effect on monotonic mechanical properties was observed. To the contrary, all three alloys experienced an increase in yield strength when pre-straining to >75% of UTS. This phenomenon has not yet been established within the literature, in particular for such high pre-strain values. This is valuable information in terms of material selection for landing gear components, since even the hardest routine landings experienced by commercial aircraft will not likely exceed >75% UTS values, however, if these components were ever placed under such extreme conditions, it's likely that this would result in an increase in yield strength, as shown.

#### 3.4. Fatigue testing +75% and 95% overload

Figure 6a–c show the tensile curves for all materials after witnessing fatigue exposure for 25% of their average fatigue life, followed by tensile overloads of 75% and 95% of their UTS, where the baseline tensile curve (without fatigue) is also shown for comparison.

Assessment of fatigue combined with overload compared against baseline tensile data leads to the following observations. Similarly to prestrain results, the UTS, yield strength and 0.2% proof stress are all seen to increase when applied with 3000 fatigue cycles and an increasing overload. In terms of UTS, changes are slight, but a similar correlation is seen, where the UTS of 300M peaks at 95% overload and AerMet100 and MLX17 peaking at 75%. Yield strength values increase more notably with increasing overload, however MLX17 witnesses a decrease in yield when exposed to fatigue +95% overload. A decrease in Youngs Modulus across all three alloys is observed with fatigue and increasing overload.

When comparing these results to pre-strain without fatigue data, as discussed in section 3.3, all three alloys show a strengthening behaviour in terms of yield, where the addition of cyclic loading and overload has resulted in greater yield strength values than that of pre-strain alone. This concurs with a number of studies in the literature where short exposure to fatigue at a moderate stress amplitude is seen to result in a hardening effect. Mocko et al has shown this effect in a number of alloys including dual phase steels and medium carbon steels, where initial fatigue exposure causes an increase in yield strength in such materials [19]. This effect is apparent for fatigue exposures of 25% and 50% of the total fatigue life, however when increased to 75%, a significant decrease in residual flow stress is observed. In this case, two subsequent phenomena are being observed; cyclic hardening during initial cycles (50% fatigue life) followed by cyclic softening with the development of fatigue damage, when approaching and exceeding 75% of the total fatigue life [20]. This is an important observation in terms of landing gear components, who's dominating loading condition is fatigue. In some other studies, however, the opposite effect has been seen to occur. Wang et al, discuss a reduction in tensile properties with the addition of pre-fatigue in as little as 20% lifetime fraction for a ferritic martensitic steel [21]. In this case, tests were conducted at temperatures of 650  $^{\circ}\text{C},$  which is believed to be a key contributory factor, whereas experimental studies carried out at room temperature, tend to report an improvement in mechanical properties.

When assessing the effect of pre-fatigue and overload on the UTS of materials, in comparison with pre-strain only data, 300M responds in a slightly different manner to AerMet100 and MLX17. Although greater than the baseline tensile value, 300M witnesses a decrease in UTS with the addition of fatigue cycling and overload, when compared to that of pre-strain alone. AerMet100 and MLX17, on the other hand, demonstrate UTS values greater than their pre-strain-only counterparts, once again, suggesting a hardening effect, with values peaking at fatigue +75% overload. It is well established that the effects of fatigue are sensitive to a materials microstructure and stress-strain characteristics can differ greatly due to influence of pre-fatigue being highly dependent on material type and loading parameters [19]. In the case of 300M, as described previously, its resultant microstructure varies from that of AerMet100 and MLX17 due to levels of retained austenite, likely meaning a variation in tensile response.

As with pre-strain only data, the addition of 3000 fatigue cycles with 75% or 95% overload, does not have a detrimental effect on any of the three materials.

#### 4. Conclusions

- The tensile mechanical properties of 300M, AerMet100 and MLX17 martensitic steels have been explored and defined in their recommended heat-treated states for landing gear application. Existing ultra-high strength steels are reported to have UTS values in the region of, or exceeding 2000MPa whereas novel corrosion-resistant high strength steel, MLX17 demonstrates a lower UTS value of ~1700MPa. Yield stresses across all three alloys are similar, being in the region of 1400MPa.
- Samples were pre-strained to 75% and 95% of their UTS values to simulate extreme tensile overload scenarios. When exposed to a 75% pre-strain, all samples demonstrated evidence of work hardening. When 95% pre-strain was used, samples either saw a very slight increase in UTS or reduced. (AerMet100) This suggests that although 95% is not detrimental, work hardening effects plateau between 75-95% for the materials assessed.
- In terms of yield strength, when pre-straining, the most significant effect is seen in 300M. When a pre-strain of 95% of its UTS is applied, the yield strength of 300M increases from 1360MPa to 1722MPa. Similar observations can be made when an initial pre-fatigue is applied plus 95% overload, where the materials yield is elevated to 1748MPa. It is likely that these variations are owed to the microstructure, with 300M exhibiting levels of retained austenite.
- Similarly to pre-strain only conditions, pre-fatigue plus overloads of 75% and 95% also results in an improvement in mechanical properties when compared to each alloys baseline tensile values. Yield strength is also seen to be greater than under pre-strain alone.
- Under all loading conditions assessed within this study, tensile
  mechanical properties are not seen to witness substantial degradation, but a general improvement in terms of yield strength and
  UTS when exposed to pre-strain or pre-fatigue plus overload. Responses vary slightly between alloys due to material
  microstructure.

#### Declarations

#### Author contribution statement

Hollie L. Cockings: Analyzed and interpreted the data; Wrote the paper.

Ben J. Cockings: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Karen M. Perkins: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

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#### Competing interest statement

The authors declare no conflict of interest.

#### Additional information

The raw data that has been used within this study is not available to share.

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