

Contents lists available at ScienceDirect

Materials Science & Engineering A



journal homepage: www.elsevier.com/locate/msea

Mechanical properties of thermally-stable, nanocrystalline bainitic steels

Check for updates

C.N. Hulme-Smith^{*}, H.K.D.H. Bhadeshia

University of Cambridge Department of Materials Science and Metallurgy, 27 Charles Babbage Road, Cambridge CB3 0FS, United Kingdom

ARTICLE INFO

Keywords: Mechanical characterization Nanostructured materials Bainite

ABSTRACT

Two novel, thermally stable bulk nanocrystalline bainitic steels were subjected to a range of mechanical tests. One alloy, containing 0.72 wt% carbon exhibited an ambient-temperature 0.2% proof strength of 1500 MPa and a fracture toughness of 64.6 MPa $m^{\frac{1}{2}}$ after the bainite transformation. The other, containing 0.45 wt% carbon and 13.2 wt% nickel, had a 0.2% proof stress of 1000 MPa and a fracture toughness of 103.8 MPa $m^{\frac{1}{2}}$. Both steels showed excellent creep resistance, with a rupture life at 450 °C and 700 MPa of 114 h and 94.8 h, respectively. Both displayed fatigue lives consistent with other steels of similar structure in the literature. After thermal exposure at 480 °C for 8 d, both steels increased in strength to 1800 MPa, and 1600 MPa, respectively. The latter steel reduced in fracture toughness to 19.6 MPa $m^{\frac{1}{2}}$. These alloys are suitable for a range of engineering applications and remain so after thermal exposure. Combined with impressive high-temperature performance, this makes the current alloys candidates for use in some elevated temperature applications.

1. Introduction

Building on earlier work, two novel bulk nanocrystalline bainitic steels (Table 1) have been developed to resist thermal decomposition [1,4]. Bulk nanocrystalline steels are well-known to possess an impressive combination of strength and toughness [2,3] and, in combination with enhanced thermal stability, this class of alloys are particularly suited to use in applications where with high demands on mechanical performance with prolonged exposure to elevated temperature, for example in gas turbine engines and power generation. The current alloys are subjected to a barrage of mechanical tests (tensile, fracture toughness, impact toughness, fatigue and creep tests) to prove their suitability for such applications.

2. Experimental methods

All mechanical tests were performed by Westmoreland Mechanical Testing and Research Ltd. of Banbury, U. K. and were in accordance to industry standards. All testpiece geometries are illustrated in supplementary figures S1–S4 and S6. In order to assess the performance of the alloys under conditions that may be expected in service, mechanical test were performed at both ambient temperature and at the elevated temperature of 450 °C. Due to equipment constraints, elevated temperature toughness experiments were performed at 150 °C. To derive the effect of heating, mechanical properties of the alloys were measured both in the as-transformed condition and after prolonged thermal

exposure (Table 2). According to the well-known Larson-Miller parameter, the tempering condition is equivalent to 60 y of exposure at $400 \degree$ C, typical of the requirements of a component in a gas turbine engine. The temperature is chosen to minimise the time of heat treatment while avoiding phase changes.

2.1. Tensile properties

Tensile tests were performed in accordance with ASTM E21-09 and using industry-standard testpieces (supplementary figure S1). A constant strain rate of 0.002 min^{-1} was used for all tests, which were run to failure.

2.2. Toughness

Toughness was assessed using both crack-tip opening displacement (CTOD) fracture toughness tests and Charpy impact tests. Testpiece geometries are shown in supplementary figures S2 and S3, respectively.

For CTOD tests, load was applied with a ratio of minimum stress to maximum stress, *R*, of 0.1. Following Dieter [17], the following dimensions apply to the current samples: a = 10.5 mm, W = 26 mm and B = 13 mm, (figure S2, Eq. (1)). Failure was deemed to have occurred when the crack-tip opening displacement crossed the 95% secant of the initial linear region of the data (Fig. 1). To form a valid measurement of $K_{\rm lc}$, conditions must be satisfied as described in the standard ASTM E399-12E3, to ensure plane strain at the crack tip

* Corresponding author.

E-mail address: cns28@cam.ac.uk (C.N. Hulme-Smith).

http://dx.doi.org/10.1016/j.msea.2017.04.110 Received 24 March 2017; Accepted 27 April 2017

Available online 03 May 2017

0921-5093/ © 2017 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY license (http://creativecommons.org/licenses/BY/4.0/).

Table 1

Composition in wt% of current alloys. All other elements ≤ 0.01 wt. %.

Alloy	С	Mn	Ni	Al	Мо	Со	Cu	Si
Alloy 1	0.72	0.02	3.41	1.38	0.21	—	—	—
Alloy 2	0.45	0.15	13.20	2.63	0.30	3.99	0.06	0.03

Table 2

Transformation conditions for mechanical testpiece blanks.

	Temperature/°C	Time/h
Austenitisation	1000	0.5
Transformation	250	24 (Alloy 1)
Tempering	480	120 (Alloy 2) 192



Fig. 1. Schematic representation of a CTOD curve and the condition taken to represent failure (black circle).

during failure. If these conditions are not met, the measured toughness is designated K_Q and is not a material property, but does allow comparison between samples. The expression for K_Q is given in Eq. (1), where P_Q is the load applied at failure an d*a*, *B* and *W* are sample dimensions, as shown in figure S2.

$$K_{Q} = \frac{P_{Q}}{W^{\frac{1}{2}B}} \left[29.6(\frac{a}{W})^{\frac{1}{2}} - 185.8(\frac{a}{W})^{\frac{3}{2}} + 655.7(\frac{a}{W})^{\frac{5}{2}} - 1017.0(\frac{a}{W})^{\frac{7}{2}} + 638.9(\frac{a}{W})^{\frac{9}{2}} \right] MPa m^{\frac{1}{2}}$$
(1)

2.3. Fatigue properties

Fatigue was assessed according to ASTM E466-07 with axial tensile loading. All tests were performed with R = 0.1 and a trapezoidal waveform (supplementary figure S4). A schematic representation of the samples is shown in supplementary figure S5. Maximum stresses were chosen below the yield stress of the material, at stresses typical of aeronautical and automotive applications. Alloy 1 was tested with peak stresses of 1200–1400MPa at ambient temperature and 800–1000MPa for tests conducted at 450 °C. Samples of Alloy 2 were tested at a peak stress of 1000–1200MPa under ambient conditions and 800 MPa at elevated temperature.

2.4. Creep properties

Creep properties were measured under a constant stress of 700 MPa at 450 °C and in accordance with ASTM 139-11. Sample geometry is shown in supplementary figure S6.

2.5. Microstructures

All microstructural investigations were performed on samples ground using silicon carbide paper, polished using diamond paste to a

Table 3

Tensile test results for Alloy 1 and Alloy 2 in as-transformed and tempered conditions. All tests performed at a constant crosshead speed of 0.002 min^{-1} .

Alloy	Temper	Test temp.	0.2% proof stress/MPa	σ _{UTS} / MPa	Failure elong. (%)	Red. of area (%)
Alloy 1	×	ambient	1432 1540	1737 1838	6.7 6.6	19.5 21.2
Alloy 1	×	450 °C	972 1035	1139 1170	27.5 27.5	88.1 84.6
Alloy 2	×	ambient	1015 1006 990 996	1435 1429 1446 1437	10.5 11.9 12.5 12.1	34.9 42.6 45.4 43.8
Alloy 2	×	450 °C	791 766	894 859	23.5 29.2	81.9 85.3
Alloy 1	1	ambient	1767 N/A	1795 1717	0.4 0.4	1.8 1.6
Alloy 2	1	ambient	1615 1591	1941 1893	1.4 1.2	3.0 3.0

 $1 \,\mu$ m finish and etched using 5% nital solution. Scanning electron microscopy was performed using a FEI NOVA NanoSEM with an accelerating voltage of 20 keV and a working distance of approximately 5 mm. No processing was used on fractographic samples.

3. Results and discussion

3.1. Tensile

The results of all tensile tests are summarised in Table 3. Alloy 1 possesses greater strength than Alloy 2 in the as-transformed bainitic condition, both at ambient temperature and at 450 °C. This is consistent with its higher carbon content and smaller grain size. Tempering also increases the 0.2% proof stress and ultimate tensile strength of both alloys, consistent with the transformation of austenite to the less-ductile ferrite and the precipitation of cementite. The 0.2% proof stress and ultimate tensile strength of Alloy 1 are consistent with other nanocrystalline steels reported in literature [9–16], as is the elongation at failure [5]. The design of the current alloys to improve thermal stability has, therefore, not compromised mechanical properties. Alloy 2 has lower strength than most nanocrystalline steels reported in literature, consistent with its lower carbon content. The reduction of area to failure is, however, greater than that reported in a conventional nanocrystalline steel by García-Mateo et al. [5]. This is consistent with the higher initial fraction retained austenite, which accommodates deformation. García-Mateo noted that 15% retained austenite remained close to the fracture surface following tensile tests [5]. This is above the percolation threshold (approximately 10% [6,7]), implying that failure occurred before austenite percolation was lost. The same study also found that raising the transformation temperature from 200 °C to 250 °C increases in the reduction of area at failure from 7% to 32%. The authors surmised that this was because the sample transformed at 250 °C had less carbon in the austenite and a wider distribution of blocky austenite grain sizes and carbon contents, leading to a wider distribution of austenite strengths and carbon contents. In the current experiments, this is analogous to the move from Alloy 1 to Alloy 2.

Fractographs of both alloys tested in the as-received condition showed extensive ductile cleavage (Fig. 2). The appearance of both alloys was very similar to figure 8.18 in Hull [8], depicting ductile cleavage in Fe–0.2 C–1.4 Mn (wt%). Alloy 1 showed ductile cleavage across almost all the fracture surface, which was predominantly flat. By contrast, only the central region of Alloy 2 showed cleavage, with a large proportion of the surface lying at 45° to the tensile axis in a classic cup-and-cone fracture (cf. figure 8.17 of [8]). This is consistent with the higher ultimate tensile strength of Alloy 1, which delays final fracture



Fig. 2. Tensile test fracture surfaces for (a) Alloy 1 and (b) Alloy 2 in the as-transformed condition. Both samples exhibit extensive ductile cleavage.

until cracks consume a large proportion of the cross sectional area; a comparatively small proportion of the cross-sectional area had to be cracked to cause catastrophic failure of Alloy 2.

Tempering at 480 °C for 8 d results in both alloys becoming stronger and less ductile (Table 3). The fracture mode of both alloys changes after tempering to exhibit areas of intergranular failure (Fig. 4), suggesting a reduction in ductility and toughness. Stress-strain curves are given in Fig. 3 from which it is apparent that Alloy 1 undergoes very little plastic deformation before fracture whereas Alloy 2 can still accommodate significant plasticity before failure, albeit an order of magnitude less than in the as-transformed condition. This is consistent with the decomposition of austenite to cementite and ferrite [9,10]. The loss of carbon from solid solution does not lead to a loss of strength as the high density of interfaces dominates the strength in the astransformed steel [37–39]. The reduction of ductility in Alloy 1 is consistent with the almost total loss of austenite due to the tempering process [1]. The magnitude of the reduction of ductility in Alloy 2 is



Fig. 3. Stress-strain curves for the elevated temperature tensile tests. All tests concluded with sample failure.



Fig. 4. Tensile test fracture surfaces for (a) Alloy 1 and (b) Alloy 2 after transformation and tempering at 480 °C for 8 d. The failure of Alloy 1 appears to have initiated close to the centre of the sample and Alloy 2 exhibits extensive inter granular failure.

surprising, given that ≥ 20 vol.% austenite persists after tempering, but is consistent with the perceived change in failure mode to quasi cleavage (Fig. 4b). No evidence was found of martensite at or below the fracture surface at any stage of the current study. However, it was found that the austenite lattice parameter decreased due to tempering [1]. This implies that carbon had left solid solution and precipitated as cementite [4]. This cementite could restrict ductility and reduce strength, and it is noticeable that the fracture surface contains a significant amount of intergranular cleavage (Fig. 4b), which is indicative of reduced strength and toughness compared to a fracture surface that exhibits entirely ductile cleavage (c.f. Fig. 2).

3.2. Toughness

Alloy 1 exhibits a toughness significantly higher than previouslyreported values of bulk nanocrystalline bainitic steels of similar strength (Table 4; cf. [9–16]). The large quantity of silicon in Alloy 1

Table 4

Compact tension results for Alloy 1 and Alloy 2. Tempering was at 480 °C for 8 d. Alloy 1 tested after tempering and Alloy 2 tested at 150 °C failed during pre-cracking.

Alloy	Tempered	Test temperature/°C	$K_{\rm Q}$ / MPa m ¹ / ₂	$K_{\rm Ic}/{\rm MPa~m^{1}_{2}}$
Alloy 1	×	ambient	60.2	
				69.0
Alloy 2	×	ambient	105.6	
			101.9	
Alloy 1	×	150	106.0	
			126.0	
Alloy 2	1	ambient	19.8	

has not, therefore, led to embrittlement. This may be explained by the lower carbon content of Alloy 1 compared to the values presented in literature, which all contained ≥ 0.78 wt% carbon. The austenite in Alloy 1 is therefore able to deform more readily and extensively as dislocations are less pinned by Cottrell atmospheres. This increase in high-stress deformation represents an energy-absorbing mechanism and will contribute to toughness.

It is also evident that as-transformed Alloy 2 is extremely tough, so much so that no valid measurements of $K_{\rm Ic}$ were possible. Future tests must use larger testpieces to ensure a valid measurement is made. However, the comparison with the $K_{\rm Q}$ of Alloy 1 suggests that Alloy 2 is significantly tougher, as is expected given the lower bulk carbon content, much higher level of nickel and higher content of retained austenite. Nickel reduces the cross-slip energy of austenite, allowing easier dislocation glide, as does spreading the deformation over a larger volume due to the greater quantity of austenite. Postponing work hardening allow easier and more extensive deformation before the onset of significant work hardening ad, ultimately, fracture.

Two samples each of both Alloy 1 and Alloy 2, tempered at 480 °C for 8 d, were sent for fracture toughness testing. All but one sample failed during pre-cracking. It was therefore decided to perform Charpy V-notch tests to gauge the toughness of the tempered alloys. The single fracture toughness measurement obtained for tempered Alloy 2 of 19 MPa $m^{\frac{1}{2}}$, is close to the fracture toughness of conventional nanocrystalline steels in the as-transformed condition [9–16], demonstrating the excellent potential of Alloy 2 for high-temperature applications. The residual toughness of Alloy 2 is likely due to the persistence of ductile austenite and the presence of nickel within that austenite, which raises the stacking fault energy. This allows easier dislocation glide and therefore mechanically stabilises the austenite [1,18–20].

Impact energies are listed in Table 5. To allow the effect of tempering to be investigated, the impact energies must be converted to the equivalent fracture toughnesses. There are several equations that may be used to compare Charpy V-notch impact test results to fracture toughness data [21]. Of these, the Rolfe-Novak-Barsom (Eq. (2), where $C_{\rm V}$ is the Charpy V-notch impact energy in Joules and other symbols have their usual meanings) provides a straightforward and well-tested conversion [22-25]. It is only possible to convert from Charpy impact energy to fracture toughness above the ductile-brittle transition temperature (DBTT). This is because fracture toughness tests are performed such that the sample fails with plane strain and in a ductile manner. If impact testing measures the absorbed energy for brittle failure, there is no correspondence between the tests. There are further restrictions on the use of the Barsom-Rolfe-Novak equation that the yield stress of the material satisfies $270 < \sigma_v/MPa < 1700$ and the measured Charpy Vnotch impact energy must lie in the range $4 < C_v/J < 82$ [21]. Both of these conditions are met for both alloys so a comparison with the fracture toughnesses of the as-transformed specimens is possible. An alternative relationship is Roberts' lower bound (Eq. (3)), which provides a more conservative estimate of $K_{\rm Ic}$ [40]. The calculated fracture toughness values are given in Table 6, using Young's moduli calculated from the elastic loading of samples prior to creep tests (Section 3.4). Both conversions predict that the toughness has decreased significantly after tempering, consistent with the loss of austenite, as observed in synchrotron X-ray diffractometry experiments

 Table 5

 Ambient temperature Charpy impact results for Alloy 1

 and Alloy 2 tested after tempering at 480 °C for 8 d.

Alloy	Impact energy/J
Alloy 1	11.5 11.5
Alloy 2	46.8 49.5

Materials Science & Engineering A 700 (2017) 714-720

Table 6

Fracture toughness estimates based on Charpy V-notch impact energies, C_V , converted using the Rolfe-Novak-Barsom equation and Roberts' lower bound. Young's moduli were derived from the elastic loading of samples at the beginning of creep tests.

Alloy	$C_{\rm V}/{\rm J}$	E/GPa	$K_{\rm Ic}$ / MPa m ¹ / ₂ (Rolfe- Novak-Barsom)	$K_{\rm Ic}$ / MPa m ¹ / ₂ (Roberts' lower bound)
Alloy 1	8.5	190	33	29
	8.5	190	33	29
Alloy 2	34.5	170	89	58
	36.5	170	92	60

on the present alloys [1].

$$K_{\rm Ic} = \left(0.228 E C_{\rm V}^{\frac{3}{2}}\right)^{\frac{1}{2}} \,\mathrm{MPa} \,\mathrm{m}^{\frac{1}{2}}$$
 (2)

$$K_{\rm Lc} = 8.47 C_{\rm V}^{0.63} \,\,{\rm MPa} \,{\rm m}^{\frac{1}{2}}$$
 (3)

3.3. Fatigue

The ambient-temperature fatigue lives (Fig. 5) of both alloys are consistent with other bulk nanocrystalline steels published in literature [26]. No fatigue limit is detected, although an identical experiment by Peet et al. [26] recorded a fatigue limit of 855 MPa in a similar bulk nanocrystalline bainitic steel, below the peak stress levels used in the current tests. The peak stresses used here were chosen to represent typical engineering applications.

The number of cycles to failure indicates that the failure mechanism is low-cycle fatigue. A modified Basquin relation is therefore used to characterise the data (Eq. (4), where σ_{max} is the peak stress, *N* is the number of cycles to failure and *a* and *b* are fitted constants). *a* and *b* are



Fig. 5. Fatigue lives for (a) Alloy 1 and (b) Alloy 2 at ambient temperature (+) and at 450 °C (\times). Horizontal lines are drawn at the yield stress and the curved lines represent the modified Basquin equation fitted to the experimental data. Points accompanied by an arrow indicates that the sample survived the entire experiment of 50,000 cycles.

refined using a Marquardt-Leverberg linear regression. An upper bound to the fatigue strength was obtained by assuming that when the peak stress exceeded the yield stress, failure would be tensile rather than fatigue.

$$\sigma_{\max} = a(2N)^{-b} \tag{4}$$

The consistency in fatigue lives shown by samples Alloy 1 tested under identical conditions (Fig. 5a) indicates that failure was not initiated at occasional large flaws, which would lead to significant scatter in the data, but at numerous small flaws or at inherent features in the material such as grain boundaries, precipitates or the surface. Conversely, Alloy 2 shows more variation between experiments conducted under the same conditions (Fig. 5b). This is consistent with the difference in processing: Alloy 1 was produced using vacuum-induction melting followed by vacuum arc remelting (VIM/VAR), which will minimise impurities and associated defects; Alloy 2 was cast using VIM only. Alloy 1 was also hot-rolled with a reduction ratio of 7:1, leading to a reduction of porosity.

In most cases, examination of the fracture surfaces revealed no obvious initiation site. Where a likely initiation site could be identified (e.g. Fig. 6) only one sample exhibited obvious flaws such as pores, inclusions or precipitates visible at or near the site, although it is possible that particular flaws did initiate fatigue failures and these particles dropped out during failure. The fracture surface (Fig. 6) of a sample of Alloy 1 tested at 450 °C shows predominantly ductile cleavage, with regions at the edges that are typical of fast fracture, the surfaces of which lie at approximately 45° to the rest of the fracture surface. These regions were the last to fail during testing. The region immediately around the initiation site shows no evidence of striations or beach marks and is approximately 100 µm wide, consistent with the observations of Peet et al. [26]. Other samples tested at ambient temperature showed similar ductile cleavage, consistent with the work of García-Mateo et al. [16], but none of them showed discernable initiation sites. Alloy 2 failed in a similar manner (supplementary figure S6) and one sample showed crack initiation at a sub-surface silica inclusion (supplementary figure S7).

The rapid failure of the samples of Alloy 1 tested at 450 °C with a peak stress of 1000 MPa is not surprising as the peak stress exceeds the yield stress (cf. Table 3) and hence leads to rapid damage accumulation. The resistance to fatigue failure at 450 °C with a peak stress of 800 MPa suggests that little damage is being accumulated. In this case, the homologous temperature is ≈ 0.4 , recovery is likely to occur, which will reduce the rate of net damage accumulation and hence extend fatigue life.

It was not possible to achieve a fatigue failure in Alloy 2 at 450 °C within the limit of 50000 cycles. The peak stress used, 800 MPa, was close to the $\sigma_{\rm UTS}$ (859 MPa, Table 3) and so it was not possible to raise the peak stress without exceeding σ_{UTS} . Failure would then be tensile



Fig. 6. Fracture surface of as-transformed Alloy 1 after fatigue testing at ambient temperature



Fig. 7. Assessed fatigue lives for Alloy 1, Alloy 2 and the alloy studied by Peet et al. [26]. The data are fitted with the modified Basquin relation (Eq. (4)) and are limited to the reported $\sigma_{\rm UTS}$. The fatigue lives of samples studied by Peet et al. were greater than the current alloys, consistent with the higher $\sigma_{\rm UTS}$ of the former. Open symbols represent samples that ran out without failure.

rather than fatigue.

Zhang et al. [27] examined low-cycle strain-controlled fatigue of bainitic steels and found that nanocrystalline bainite (called "low temperature bainite" by Zhang et al.) exhibited slower fatigue crack growth than lower and upper bainite formed in the same alloy, which was attributed to a finer grain size and larger misorientation between adjacent grains, as measured by EBSD. The authors determined that these factors lead to more rapid blunting of fatigue cracks and, hence, slower crack growth.

Peet et al. [26] obtained a longer fatigue life than either of the current alloys (Fig. 7), consistent with the alloys of Peet et al. exhibiting a higher $\sigma_{\rm UTS}$ [16,26]. Both of the current alloys exhibited large-scale ductile rupture, as was noted by García-Mateo et al. [16] in high-carbon steel. The consensus in literature is that the fatigue properties of nanostructured steel are promising and the current study has found that the fatigue lives of the current alloys are consistent with those previously reported.

3.4. Creep

There are no reported creep results for nanocrystalline bainitic steels. This is because they were not intended originally for elevated temperature service and decomposed upon heating [9,28]. It is only with the production of more thermally-stable alloys that creep data may be usefully assessed. It is apparent that Alloy 1 has superior creep life to Alloy 2 under the test conditions (Table 7, Fig. 8). Both alloys compare favourably to existing steels. For example, Jitsukawa et al. [29] collated an extensive database of 9 Cr-1 Mo (wt%) steels and reduced-activation martensitic steels for nuclear pressure vessels and found that stress levels of approximately 400 MPa corresponded to a creep life consistent with a Larson-Miller parameter of 20.5 (evaluated as 0.001 $T(26.4 + \log t_r)$ where T is the test temperature in Kelvin and t_r is the creep rupture time in seconds). The current alloys exhibit a creep life of the same Larson-Miller parameter at a stress level of 700 MPa.

Table 7				
Creep rupture life of current	t alloys under	r 700 MPa	at 45	0 °C.

Alloy	Alloy 1		Alloy 2	
Creep rupture life/h	120	107	95.9	93.6



Fig. 8. Creep strain curves measured under 700 MPa constant stress at 450 °C. All tests ended with sample failure. Both alloys exhibit a large tolerance for damage, as evidenced by the long interval between the time for minimum creep rate to the time of failure.



Fig. 9. Creep rupture strength at 100 h of current alloys (black circle) and other alloys taken from the ASM Handbook [30]. Only maraging steels and nickel superalloys, both of which are appreciably more expensive than the current alloys, are significantly more creep resistant.

The ASM handbook on heat-resistant materials lists common classes of steel and selected other materials (Fig. 9) [30]. The current alloys survive approximately 100 h at 450 °C (823 K) under a constant stress of 700 MPa. This is competitive with 12 wt% Cr steels and is only outperformed by nickel alloys and maraging steels. Both of these alloy systems are orders of magnitude more expensive than nanocrystalline bainitic steel [31–33].

While the period of primary creep (~ 10 h) and the time at which the minimum creep strain rate is observed (~20 h in Alloy 1 and ~30 h in Alloy 2) are small in both alloys, the time between minimum creep rate and failure is long. This suggests that the current alloys are able to tolerate damage accumulation well, in a manner consistent with creepresistant martensitic steels [34]. Nabarro and de Villiers [35] describe primary creep as consisting of grain boundary sliding and/or plastic deformation of grains with high Schmidt factors, resulting in work hardening and a redistribution of load to other less-favourably oriented grains. In the current case, the vast majority of the grain boundaries are between bainitic ferrite and untransformed parent austenite, which are semi-coherent. The atomic correspondence across the interface makes grain boundary sliding extremely unlikely. It may, however, be possible for the incoherent interfaces at prior austenite grain boundaries to slide. At the test temperature (450 °C), the applied stress (700 MPa) is approximately 70% of the 0.2% proof stress of Alloy 1 and 90% of that for Alloy 2 (Table 3). This suggests that the resolved shear stress in favourably-oriented grains could easily exceed that required for dis-



Fig. 10. (a) creep voids in the elongated region of a failed creep specimen of Alloy 2; (b) voids overlaid with the locations of the prior austenite grain boundaries. All of the voids appear to lie on such boundaries and most lie on triple points, as is classically expected for creep damage [35].

location glide and the material could, therefore, plastically deform in some grains. Since this initial deformation is likely to be confined to the ductile austenite, and the grain size is small, strain will be localised and work hardening is liable to be rapid.

Nabarro and de Villiers note that fine grain sizes are detrimental to resistance to both grain boundary sliding and diffusional creep [35]. However, in nanostructured bainite, the vast majority of grain boundaries are between retained austenite films and bainitic ferrite. These are semi-coherent boundaries and contain a well-ordered array of misfit dislocations. These boundaries will not act as efficient sources or sinks of vacancies as is the case for incoherent boundaries. Prior austenite grain boundaries are incoherent and can contribute to creep, but these are found infrequently in the structure, at a density associated with conventional grain sizes. Thus it is possible to obtain the strengthening from a fine grain structure without the penalties associated with short diffusion paths and easy grain boundary sliding. While the temperature and stress used in the current tests make it very likely that dislocation creep will be the dominant mechanism, vacancy flux is still required to allow the dislocations to overcome obstacles and mediate deformation. The use of semi coherent and coherent boundaries to pin dislocations in creep-resistant "coherent hierarchical precipitate" strengthened ferritic steels was reported by Song et al. [36] and is discussed in the case of a steel containing intermetallic precipitates of TiAl and Ti₃Al by Nabarro and de Villiers [35], who note that semi-coherent boundaries contain networks of dislocations. These misfit dislocations could act as sources and sinks for vacancies due to climb of their edge components, but this is only significant for the late stages of deformation, with the dislocations anchored at the interfaces during the initial stages of creep to



Fig. 11. Voids in a failed creep specimen of Alloy 1. Although they do not clearly lie on prior austenite grain boundaries, the voids are typical of those found immediately below a creep fracture surface [35].

maintain the semi-coherent nature of the boundary. This gives rise to one possible explanation for the extensive creep life after the minimum strain rate occurs: the creep strain rate gradually increases as deformation occurs and progressively more misfit dislocations begin to undergo climb. Furthermore, the use of ordered intermetallics noted by Nabarro and de Villiers could be replicated in Alloy 2 by tempering to produce a distribution of NiAl precipitates, analogous to the TiAl precipitates studied previously [35].

The appearance of a section of failed creep testpieces of both Alloy 2 and Alloy 1 contains severely elongated grain structure near the fracture surface, together with large numbers of voids. The voids in Alloy 2 lie predominantly perpendicular to the tensile axis and clearly follow prior austenite grain boundaries (Fig. 10). The voids in Fig. 11 do not obviously follow grain boundaries, as is seen in Alloy 2, but are otherwise typical of creep failure (e.g. figure 8.22 of [35]).

4. Conclusions

Two nanocrystalline bainitic alloys have been subject to a range of mechanical tests and found to be comparable to other alloys of the same structure. The fact that the current alloys also exhibit improved thermal stability compared to previously-published alloys of this type makes them suitable for use in elevated-temperature engineering applications for the first time. Using the thermal stability, the creep properties of the alloys was measured and found to be competitive with other creepresistant steels, despite the high density of interfaces and the comparatively low cost of the current alloys. These are the first published creep data for this alloy class.

Acknowledgements

The authors would like to thank Rolls-Royce plc and the Engineering and Physical Sciences Research Council for funding the work (grant RG64823) and Professors Lindsay Greer and Mark Blamire for the provision of laboratory facilities.

Appendix A. Supplementary data

Supplementary data associated with this article can be found in the

online version at http://dx.doi.org/10.1016/j.msea.2017.04.110.

References

- C.N. Hulme-Smith, S.W. Ooi, H.K.D.H. Bhadeshia, Mater. Metall. Trans. A, Article in press 2017.
- [2] H.K.D.H. Bhadeshia, Solid-Solid Phase Transformations in Inorganic Materials, TMS, May–June 2005, Phoenix, Arizona, U. S. A., pp. 469–484.
- [3] C. García-Mateo, F.G. Caballero, Understanding the Mechanical Properties of Nanostructured Bainite in Mahmood Aliofkhazraei (ed.) Handbook of Mechanical Nanostructuring, Wiley 2015, pp. 35–65.
- [4] C.N. Hulme-Smith, I. Lonardelli, M.J. Peet, A.C. Dippel, H.K.D.H. Bhadeshia, Scipta Mater. 69 (2013) 191–194.
- [5] C. García-Mateo, F.G. Caballero, T. Sourmail, M. Kuntz, J. Cornide, V. Smanio, R. Elvira, Mater. Sci. Eng. A 549 (2012) 185–192.
- [6] H.K.D.H. Bhadeshia, Mater. Sci. Eng. A 481–482 (2008) 36–39.
- [7] H.K.D.H. Bhadeshia, Proc. R. Soc. A Math. Phys. 466 (2009) 3–18.
- [8] D. Hull, Fractography, Cambridge University Press, Cambridge, U. K., 1999.
 [9] C. García-Mateo, M.J. Peet, F.G. Caballero, H.K.D.H. Bhadeshia, Mater. Sci. Technol. 20 (2004) 814–818.
- [10] M.J. Peet, Transformation and tempering of low-temperature bainite, (Ph.D. thesis), University of Cambridge, Cambridge, U. K., 2010.
- [11] F.G. Caballero, H.K.D.H. Bhadeshia, K.J.A. Mawella, D.G. Jones, P.M. Brown, Mater. Sci. Technol. 18 (2002) 279–284.
- [12] P.M. Brown, D.P. Baxter, Materials Science and Technology 2004, New Orleans, Louisiana, U. S. A., 2004, pp. 433–438.
- [13] C. García-Mateo, F.G. Caballero, ISIJ Int. 45 (2005) 1736-1740.
- [14] C. García-Mateo, F.G. Caballero, H.K.D.H. Bhadeshia, Mater. Sci. Forum 500–501 (2005) 495–502.
- [15] F.G. Caballero, J. Chao, J. Cornide, C. García-Mateo, J. Santofima, C. Capdevila, Mater. Sci. Eng. A 525 (2009) 87–95.
- [16] C. García-Mateo, T. Sourmail, F.G. Caballero, V. Smanio, M. Kuntz, A. Leiro,
- E. Vuorinen, R. Elvira, T. Teeri, Mater. Sci. Technol. APMS Spec. Issue (2013) 1–8.
 [17] G.E. Dieter, Mechanical Metallurgy, SI Metric edition, McGraw-Hill Book Company Limited, U. K., 1988.
- [18] B. Avishan, S. Yszdani, S.H. Nedjad, Mater. Sci. Eng. A 548 (2012) 106-111.
- [19] L. Vitos, J.-O. Nillson, B. Johannson, Acta Mater. 54 (2006) 3821–3826.
- [20] S. Curtze, V.-T. Kuokkala, A. Oikari, J. Talonen, H. Hänninen, Acta Mater. 59 (2011) 1068–1076.
- [21] P.R. Sreenivasan, Eng. Fract. Mech. 75 (2008) 5229-5245.
- [22] J.M. Barsom, S.T. Rolfe, ASTM STP 466 pp. 281-302.
- [23] J.M. Barsom, S.T. Rolfe, Eng. Fract. Mech. 2 (1971) 341-357.
- [24] B. Ule, A. Ažman, A. Lagoja, A. Jesenice, Kovine, zlitine, tehnologije 27 (1993) pp. 283–288.
- [25] J. A. Kapp, J. A. Underwood, Correlation between fracturetoughness, Charpy Vnotch impact energy, andyield strength for ASTM A723 steel, U. S. Army MemorandumReport ARCCB-MR-92008, 1992.
- [26] M.J. Peet, P. Hill, M. Rawson, S. Wood, H.K.D.H. Bhadeshia, Mater. Sci. Technol. 27 (2011) 119–123.
- [27] F.C. Zhang, X.Y. Long, J. Kang, D. Cao, B. Lv, Mater. Des. 94 (2016) 1–8.
- [28] A. Saha Podder, H.K.D.H. Bhadeshia, Mater. Sci. Eng. A 527 (2010) 2121–2128.
 [29] S. Jitsukawa, M. Tamura, B. van der Schaaf, R.L. Klueh, A. Alamo, C. Petersen,
- M. Schirra, P. Spaetig, G.R. Odette, A.A. Tavassoli, K. Shiba, A. Kohyama, A. Kimura, J. Nucl. Mater. 307–311 (2002) 179–186.
- [30] ASM Speciality Handbook: Heat-Resistant Materials, ASM International, Materials Park, Ohio, U. S. A., 1997.
- [31] C. García-Mateo, F.G. Caballero, T. Sourmail, V. Smanio, C. García De Andrés, Int. J. Mater. Res. 105 (2014) 725–734.
- [32] H.K.D.H. Bhadeshia, Steels: Microstructure and Properties, Elsevier Ltd, Oxford, U. K, 2006.
- [33] CES EduPack, Granta Design Ltd, Cambridge, U. K., 2011.
- [34] K. Maruyama, K. Sawada, J.-I. Koike, ISIT Int. 41 (2001) 641-653.
- [35] F.R.N. Nabarro, H.L. de Villiers, The Physics of Creep, Taylor & Francis, London, U. K, 1995.
- [36] G. Song, Z. Sun, L. Li, X. Xu, M. Rawlings, C.H. Liebscher, B. Clausen, J. Poplawsky, D.N. Leonard, S. Huang, Z. Teng, C.T. Liu, M.D. Asta, Y. Gao, D.C. Dunand, G. Ghosh, M. Chen, M.E. Fine, P.K. Liaw, Sci. Rep. (5) (2015) (article no. 16327).
- [37] G. Langford, M. Cohen, Metall. Trans. 1 (1970) 1478–1480.
- [38] H.K.D.H. Bhadeshia Proceedings Roy. Soc. A 466 (2009) pp. 3–18.
- [39] F.B. Pickering, Physical metallurgy and the design of steels, first edition, Applied
- Science Publishers, London, U. K, 1978.
- [40] R. Roberts, C. Newton, Welding Research Council Bulletin 265, Welding Research Council, New York, U. S. A., 1981.