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Technical Note

Tensile Ductility of Nanostructured Bainitic Steels: Influence of Retained Austenite Stability

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Abstract: High silicon (>1.5%) steels with different compositions were isothermally transformed to bainite at 220 and 250 °C to produce what is often referred to as nanostructured bainite. Interrupted tensile tests were carried out and the retained austenite was measured as a function of strain. Results were correlated with tensile ductility. The role of retained austenite stability is remarkably underlined as strongly affecting the propensity to brittle failure, but also the tensile ductility. A simple quantitative relationship is proposed that clearly delimitates the different behaviours (brittle/ductile) and correlates well with the measured ductility. Conclusions are proposed as to the role of retained austenite fraction and the existence of a threshold value associated with tensile rupture.

Keywords: high carbon steels; nanobainite; low temperature bainite; tensile ductility; retained austenite stability; transformation induced plasticity (TRIP)

1. Introduction

Bainitic microstructures formed at low temperatures (350 °C or less) have received a considerable amount of attention in the recent years [1–14]. These microstructures are obtained in relatively high carbon steels (0.6–1.2 wt %, although the concept can be extended to lower carbon contents) through isothermal transformation over durations ranging from 10 to over 100 h [5,11,15]. They consist of ultrafine bainitic laths (typical width under 50 nm) surrounded by retained austenite [3,11]. Interestingly, the initial mechanical properties [2] were at best on par with those of quenched and tempered high strength spring steels [16], but were later improved to reach an unprecedented 21% elongation for over 2.1 GPa in tensile strength [10,11].

From a microstructural point of view, the yield and tensile strength of these materials have been shown to be reasonably well correlated to the parameter V_β / t_β where V_β is the volume fraction of bainitic ferrite (the rest normally being retained austenite) and t_β the average lath thickness [10,11,17]. However, tensile ductility has recently been shown to exhibit largely different values for microstructures exhibiting reasonably similar retained austenite fraction and bainitic ferrite lath thickness, as shown in Table 1, after [12].

Table 1. Tensile elongation for an identical material (1C-2.5Si wt %) transformed at 220 or 250 °C, after [12].

Reference in [12]	UTS, MPa	TE, %	$\gamma_{res} = 1 - V_\beta$, %	t_β, nm
1CSi-220	~2070	7	34	28
1CSi-250	~2200	21	36	28

UTS is Ultimate tensile strength; TE is Total elongation; γ_{res} is the volume fraction of retained austenite; V_β is the volume fraction of bainitic ferrite; t_β is the average thickness of the bainitic ferrite laths.

Earlier work on the factors controlling the ductility of nanostructured bainite has insisted on the role of retained austenite fraction [18,19] and suggested the existence of an optimum value to achieve the maximum ductility. More recent work, based on measurements of retained austenite content before tensile tests and calculated evolutions, proposed that the stability of retained austenite would influence the material ductility, and indicated that there could be an optimum stability [20]. Data from this same publication were later re-interpreted to propose the existence of a percolation mechanism, whereby ductility was imparted by a percolating network of retained austenite in the matrix of bainitic ferrite, and fracture occurred at an approximately constant volume fraction of 10% retained austenite [17]. Also recently, a model for stress-assisted martensite formation was proposed by the present authors [21] and was found to provide a reasonable agreement with the experiment, though this approach does not provide indication as to the causes of early tensile failures.

The present work is concerned with further investigating this hypothesis through the use of interrupted tensile tests. This allows actual measurement of the retained austenite content as a function rather than estimated values to be used.

2. Materials and Methods

2.1. Materials

Three materials were used for the present investigation, with compositions as indicated in Table 2. References indicate both the carbon and silicon content. Both 06C-1.5Si and 1C-2.5Si were produced industrially as ingots, then hot-rolled to 120 mm (0.6C-1.5Si) or 35 mm (1C-2.5Si) bars. The 1C-1.5Si steel was manufactured using a vacuum induction furnace to obtain an approximately 35 kg ingot. After cooling to room temperature, the ingot was re-heated to 1150 °C and forged to a 40 mm bar. Prior to machining, all steels were annealed for 2 h at 700 °C. Chemical composition was determined on the hot-rolled or forged bars using optical emission spectrometry and combustion analysis (LECO).

Table 2. Chemical composition (wt %) for the three steels used in the present investigation, as determined using optical emission spectrometry and combustion (LECO) analysis.

Reference	C	Si	Mn	Cr	Mo	V
0.6C-1.5Si	0.67	1.67	1.32	1.73	0.15	0.12
1C-1.5Si	1.05	1.60	0.74	1.05	0.07	-
1C-2.5Si	0.99	2.47	0.74	0.97	0.03	-

2.2. Heat-Treatment, Tensile Testing and Retained Austenite Measurements

Tensile specimens, 6 or 8 mm in diameter, were manufactured from the hot-rolled bars, using material taken at mid-radius of the latter. They were initially machined with 0.3–0.5 mm additional thickness then heat-treated. The heat-treatments consisted of austenitising in a first salt bath or in a conventional furnace, followed by rapid cooling in a salt bath, to the isothermal transformation temperature. Both austenitising and isothermal transformation parameters varied. Austenitising was carried out for 1 h at temperatures between 860 and 1050 °C, while the temperature for isothermal transformation varied between 220 and 250 °C. The duration for isothermal holding was determined

from measurements in a Baehr dilatometer and varied depending on the material and austenitising. For convenience, relevant heat-treatment parameters are included in the specimen reference. As an example, 1C-2.5Si-1050-250 (16 h) refers to material 1C-2.5Si austenitised at 1050 °C for one hour and isothermally transformed at 250 °C for 16 h; austenitising duration is not indicated as it was kept constant (1 h). Following heat-treatment, specimens were hard-machined to their final dimensions.

Conventional tensile tests were carried out using three to five specimens. Once yield strength, universal tensile strength and tensile elongation were known, interrupted tensile tests were carried out at selected values of plastic strain in the uniform elongation domain (so as to ensure absence of necking and non-uniform strain distribution in the specimens). Retained austenite measurements were carried out on both the tensile specimens' grip (reference value) and on transverse sections from the gauge length (value after destabilization by plastic strain).

For these experiments, samples were machined, ground and polished with 1 μm diamond paste, and then subjected to several cycles of etching and polishing to obtain an undeformed surface; finally, the samples were polished with colloidal silica. X-ray diffraction measurements were performed by means of a Bruker AXS D8 diffractometer equipped with a Co X-ray tube and Goebel mirror optics to obtain a parallel and monochromatic X-ray beam. Operational parameters and the procedure for obtaining the austenite content and composition are described elsewhere [22,23].

3. Results

Tensile tests exhibited two different behaviors which are illustrated in Figure 1. For specimens breaking in a brittle manner, both ultimate tensile strength (UTS) and elongation varied significantly (up to 800 MPa for the maximum stress and 4%–5% for the maximum elongation) in the three to five tests carried out on each identical condition, the maximum values were taken. For specimens breaking after necking, the reproducibility was typically within ±15 MPa for the UTS and ±0.7% for the elongation.

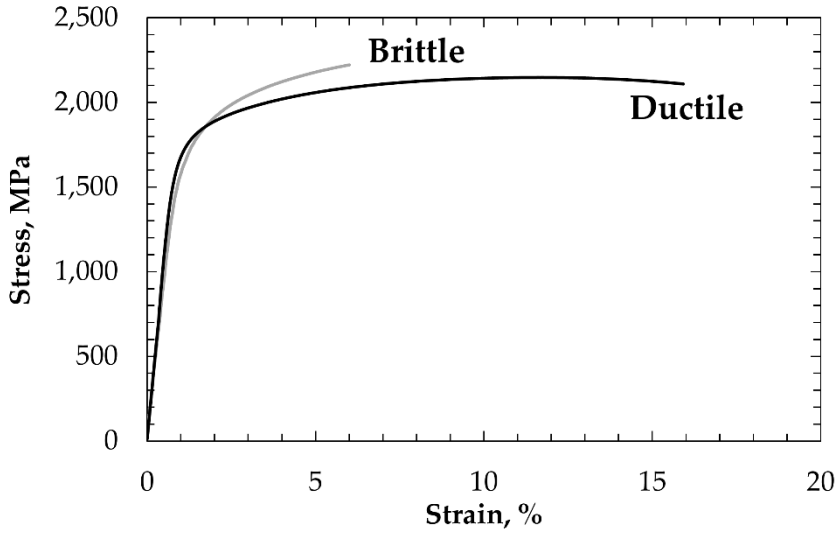

Figure 1. Example of the two different behaviours (brittle and ductile) identified on engineering stress–strain curves. Brittle behaviour may lead to higher maximum stress though the reproducibility is poor.

Tensile data for all investigated conditions are summarized in Table 3, together with retained austenite content as measured in the grip. As shown, UTS varied between 2.0 and 2.2 GPa, with elongation as high as 17%. As already reported [10,11], such results are exceptional in the combination of strength and ductility that is achieved. It is also clear that retained austenite content alone does not

correlate directly with tensile elongation, as elongations around 5% and above 10% can be found for both ~20% or ~40% retained austenite contents.

Table 3. Initial retained austenite content and tensile properties for all conditions investigated. The notation for the reference is explained in the text. * indicate brittle behaviour with no true UTS value and variable maximum elongation (the highest value of all tests is then given).

Reference	$V_{\gamma,0}$, %	$YS_{0.2\%}$, MPa	UTS, MPa	TE, %	k
0.6C-1.5Si-890-250-16h	18	1448	1990	14.3	0.087
0.6C-1.5Si-890-220-22h	22	1246	2236 *	4.7 *	0.217
0.6C-1.5Si-950-250-22h	23	1404	1990	14.4	0.068
0.6C-1.5Si-950-220-40h	24	1295	2221	8.9	0.127
H0.6C-1.5Si-890-220-22h	21	1193	2158 *	5.8 *	0.195
1C-2.5Si-950-220-22h	43	1675	2185 *	3.9 *	0.203
1C-2.5Si-950-220-70h	33	1921	2277 *	5.7 *	0.181
1C-2.5Si-950-250-16h	37	1738	2106	16.8	0.058
1C-2.5Si-950-250-40h	35	1785	2101	15.8	0.078
1C-2.5Si-1050-220-40h	41	1768	2195 *	2.6 *	0.553
1C-2.5Si-1050-250-25h	34	1676	2088	14.9	0.048
1C-1.5Si-950-220-22h	40	1192	2063 *	3.0 *	1.009
1C-1.5Si-950-250-16h	33	1740	2170	10.7	0.130

$V_{\gamma,0}$ is the retained austenite content as measured in the grip; UTS is Ultimate tensile strength; TE is Total elongation; k is the constant in Equation (1), for a plastic strain expressed in %.

In an attempt to quantify the relationship between retained austenite stability and tensile ductility, the results were represented as per the following relationship [24]:

$$\ln(V_{\gamma,0}) - \ln(V_{\gamma}) \propto k\varepsilon_p \quad (1)$$

where $V_{\gamma,0}$ is the initial retained austenite content as measured in the specimen grip, and V_{γ} the retained austenite in the gauge length after application of a true plastic strain of ε_p. Some results are illustrated as an example in Figure 2. For all conditions investigated, the value of k was estimated using linear regression (throughout, this value is given for ε_p in percent).

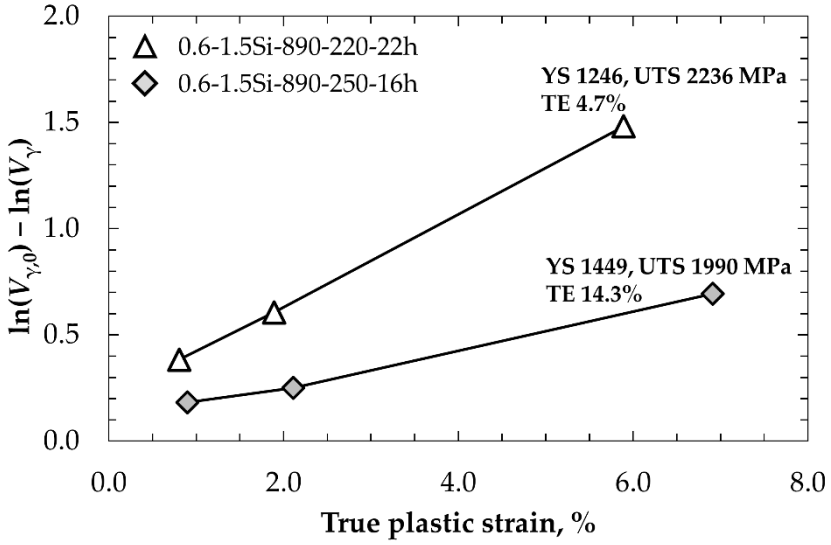

Figure 2. Destabilisation of retained austenite as a function of true plastic strain for two selected conditions.

Figure 3 shows the tensile elongation as a function of k for all conditions investigated. It is worth underlining that the corresponding dataset is for three different materials with a variety of

heat-treatments. A first and clear correlation is that relating high values of k (rapid mechanical destabilisation of retained austenite with increasing strain) with brittle behaviour. Indeed, all conditions for which k values of more than ~0.2 were measured, led to brittle fracture during full tensile tests. Inversely, below that threshold, there appears to be a direct correlation between improved mechanical stability (as measured through k) and tensile ductility.

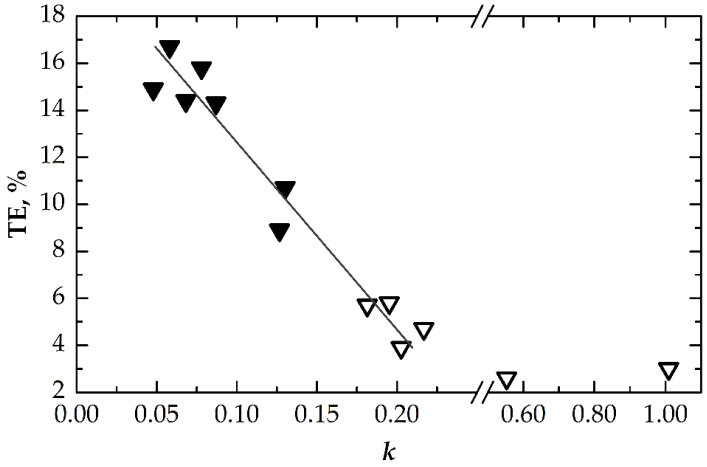

Figure 3. Tensile elongation as a function of the value of k for all conditions investigated. Hollow symbols are for brittle ruptures; full symbols are for ductile ruptures.

In addition to measurements carried out on specimens tested within the uniform elongation domain, retained austenite content was sometimes estimated on surfaces directly underneath the rupture surface using the first specimens having undergone full tensile tests. These measurements were associated with a very large texture uncertainty and are therefore to be taken with caution; they were nevertheless frequently below 10% (e.g., 4% for 1C-2.5Si-950-220-22h, 5% for 1C-2.5Si-950-250-40h). More reliably, a number of measurements within the uniform elongation domain yielded retained austenite content below 10% (0.6C-1.5Si-890-250-16h, γ_{res} 9% for 8% deformation; 0.6C-1.5Si-890-220-22h, γ_{res} 5% for 7% deformation).

4. Discussion and Conclusions

As discussed earlier, it has been proposed that the tensile ductility of bainite formed at low temperatures is correlated with the amount of retained austenite initially available, and limited to a percolation threshold, below which further plastic deformation is no longer possible [17].

The present data provide two important results. First, and as can be seen in Table 3, there is no correlation between initial retained austenite content and tensile ductility. In fact, retained austenite contents of 35%–40% can be associated with "brittle" behavior (1C-2.5Si-950-220-22h, 1C-2.5Si-1050-220-40h, 1C-1.5Si-950-220-22h) but also with very good tensile ductility (1C-2.5Si-950-250-16h). On the contrary, the approximate quantification of retained austenite stability via k exhibits an excellent correlation with tensile ductility. Furthermore, results suggest the existence of a critical value of k, beyond which brittle behavior cannot be avoided (low retained austenite stability). Below this value, the results suggest a continuous benefit in increasing retained austenite stability to enhance tensile ductility. In particular, the present results do not provide evidence that these microstructures may exhibit "excessive" austenite stability as suggested in earlier publications [22].

Second, the above results do not confirm the suggested existence of a threshold retained austenite content [17], below which ductile deformation is no longer possible.

Interestingly, poor ductility (high k values) was largely associated with transformation at 220 °C, whereas transformation at 250 °C tended to systematically provide ductile behavior (Table 3).

A detailed investigation (SEM, TEM, 3D-APT) of the potential origins of this difference will be published separately.

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Conflicts of Interest: The authors declare no conflict of interest.

References

1. Caballero, F.G.; Bhadeshia, H.K.D.H.; Mawella, K.J.A.; Jones, D.G.; Brown, P. Design of novel high strength bainitic steels: Part 1. *Mater. Sci. Technol.* **2001**, *17*, 512–516. [CrossRef]
2. Caballero, F.G.; Bhadeshia, H.K.D.H.; Mawella, K.J.A.; Jones, D.G.; Brown, P. Design of novel high strength bainitic steels: Part 2. *Mater. Sci. Technol.* **2001**, *17*, 517–522. [CrossRef]
3. Caballero, F.G.; Bhadeshia, H.K.D.H.; Mawella, K.J.A.; Jones, D.G.; Brown, P. Very strong low temperature bainite. *Mater. Sci. Technol.* **2002**, *18*, 279–284. [CrossRef]
4. Garcia-Mateo, C.; Caballero, F.G.; Bhadeshia, H.K.D.H. Development of hard bainite. *ISIJ Int.* **2003**, *43*, 1238–1243. [CrossRef]
5. Garcia-Mateo, C.; Caballero, F.; Bhadeshia, H. Low temperature bainite. *J. Phys. IV* **2003**, *112*, 285–288. [CrossRef]
6. Caballero, F.G.; Bhadeshia, H.K.D.H. Very strong bainite. *Curr. Opin. Solid State Mater. Sci.* **2004**, *8*, 251–257. [CrossRef]
7. Wang, T.S.; Li, X.Y.; Zhang, F.C.; Zheng, Y.Z. Microstructures and mechanical properties of 60Si2CrVa steel by isothermal transformation at low temperature. *Mater. Sci. Eng. A* **2006**, *438–440*, 1124–1127. [CrossRef]
8. Soliman, M.; Palkowski, H. Ultra-fine bainite structure in hypo-eutectoid steels. *ISIJ Int.* **2007**, *47*, 1703–1710. [CrossRef]
9. Timokhina, I.B.; Beladi, H.; Xiong, X.Y.; Adachi, Y.; Hodgson, P.D. Nanoscale microstructural characterization of a nanobainitic steel. *Acta Mater.* **2011**, *59*, 5511–5522. [CrossRef]
10. Garcia-Mateo, C.; Caballero, F.G.; Sourmail, T.; Kuntz, M.; Cornide, J.; Smanio, V.; Elvira, R. Tensile behaviour of a nanocrystalline bainitic steel containing 3 wt % silicon. *Mater. Sci. Eng. A* **2012**, *549*, 185–192. [CrossRef]
11. Sourmail, T.; Caballero, F.G.; Garcia-Mateo, C.; Smanio, V.; Ziegler, C.; Kuntz, M.; Elvira, R.; Leiro, A.; Vuorinen, E.; Teeri, T. Evaluation of potential of high si high c steel nanostructured bainite for wear and fatigue applications. *Mater. Sci. Technol.* **2013**, *29*, 1166–1173. [CrossRef]
12. Sourmail, T.; Smanio, V.; Ziegler, C.; Heuer, V.; Kuntz, M.; Caballero, F.G.; Garcia-Mateo, C.; Cornide, J.; Elvira, R.; Leiro, A.; et al. *Novel Nanostructured Bainitic Steel Grades to Answer the Need for High-Performance Steel Components (Nanobain)*; European Commission: Luxembourg, Luxembourg, 2013.
13. Garbarz, B.; Burian, W. Microstructure and properties of nanoduplex bainite–austenite steel for ultra-high-strength plates. *Steel Res. Int.* **2014**, *85*, 1620–1628. [CrossRef]
14. Soliman, M.; Palkowski, H. Development of the low temperature bainite. *Arch. Civ. Mech. Eng.* **2016**, *16*, 403–412. [CrossRef]
15. Garcia-Mateo, C.; Caballero, F.G.; Bhadeshia, H.K.D.H. Acceleration of low-temperature bainite. *ISIJ Int.* **2003**, *43*, 1821–1825. [CrossRef]
16. Tump, A.; Brandt, R. Graded high-strength spring-steels by a special inductive heat Treatment. *IOP Conf. Ser. Mater. Sci. Eng.* **2016**, *118*, 012021. [CrossRef]
17. Bhadeshia, H.K.D.H. Nanostructured bainite. *Proc. R. Soc. A* **2010**, *466*, 3–18. [CrossRef]
18. Sandvik, B.P.J.; Navalainen, H.P. Structure-property relationships in commercial low-alloy bainitic-austenitic steel with high strength, ductility, and toughness. *Met. Technol.* **1981**, *8*, 213–220. [CrossRef]
19. Bhadeshia, H.K.D.H. *Bainite in Steels. Transformations, Microstructure and Properties*, 2nd ed.; Institute of Materials, Minerals and Mining: London, UK, 2001.

20. Garcia-Mateo, C.; Caballero, F.G. The role of retained austenite on tensile properties of steels with bainitic microstructures. *Mater. Trans. JIM* **2005**, *46*, 1839–1846. [CrossRef]
21. Morales-Rivas, L.; Garcia-Mateo, C.; Kuntz, M.; Sourmail, T.; Caballero, F.G. Induced martensitic transformation during tensile test in nanostructured bainitic steels. *Mater. Sci. Eng. A* **2016**, *662*, 169–177. [CrossRef]
22. Garcia-Mateo, C.; Caballero, F.G.; Chao, J.; Capdevila, C.; de Andres, C.G. Mechanical stability of retained austenite during plastic deformation of super high strength carbide free bainitic steels. *J. Mater. Sci.* **2009**, *44*, 4617–4624. [CrossRef]
23. Avishan, B.; Garcia-Mateo, C.; Yazdani, S.; Caballero, F.G. Retained austenite thermal stability in a nanostructured bainitic steel. *Mater. Charact.* **2013**, *81*, 105–110. [CrossRef]
24. Sherif, M.Y.; Mateo, C.G.; Sourmail, T.; Bhadeshia, H. Stability of retained austenite in trip-assisted steels. *Mater. Sci. Technol.* **2004**, *20*, 319–322. [CrossRef]