



# Light and electron microscopy of microstructure and fractography of an ultrahigh-strength martensitic steel

Shi, X., Wang, W., Ye, W., Sha, W., Shan, Y., Shen, M., & Yang, K. (2014). Light and electron microscopy of microstructure and fractography of an ultrahigh-strength martensitic steel. Microscopy and Analysis, 28(1), 7-10.

Published in: Microscopy and Analysis

**Document Version:** Peer reviewed version

#### **Queen's University Belfast - Research Portal:**

Link to publication record in Queen's University Belfast Research Portal

#### **Publisher rights**

This is the accepted version of the following article: Shi, X, Wang, W, Ye, W, Sha, W, Shan, Y, Shen, M & Yang, K 2014, 'Light and electron microscopy of microstructure and fractography of an ultrahigh-strength martensitic steel' Microscopy and Analysis, vol 28, no. 1, pp. 7-10., which has been published in final form at http://www.microscopy-analysis.com/magazine/issues/light-and-electron-microscopy-microstructure-and-fractography-ultrahigh-strength.

#### **General rights**

copyright owners and it is a condition of accessing these publications that users recognise and abide by the legal requirements associated with these rights. Copyright for the publications made accessible via the Queen's University Belfast Research Portal is retained by the author(s) and / or other

Take down policy The Research Portal is Queen's institutional repository that provides access to Queen's research output. Every effort has been made to ensure that content in the Research Portal does not infringe any person's rights, or applicable UK laws. If you discover content in the Research Portal that you believe breaches copyright or violates any law, please contact openaccess@qub.ac.uk.

# Microstructure and fractography of an ultra-high strength steel

Xianbo Shi<sup>1,2</sup>, Wei Wang<sup>1</sup>, Wei Ye<sup>1</sup>, Wei Sha<sup>3</sup>, Yiyin Shan<sup>1,\*</sup>, Minggang Shen<sup>2</sup>, Ke Yang<sup>1</sup>

Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
University of Science and Technology Liaoning, Anshan 114051, China

3. Metals Research Group, School of Planning, Architecture & Civil Engineering, Queen's University Belfast, Belfast BT9

## 5AG, UK

**Biography:** Wei Sha obtained a BEng at Tsinghua University in 1986. He was awarded in 1992 a PhD by Oxford University and in 2009 a DSc by Queen's University Belfast. He previously worked at Imperial College and Cambridge University. He is presently Professor of Materials Science, with research interests in microscopy.

**Abstract:** Microstructure, tensile properties and fractography have been examined, in the oil quenched samples of a low-alloy ultra-high strength 4340 steel. Intergranular fracture was revealed to locate at the fracture origin. However, neither the quenched Charpy V-notched impact samples nor the tempered tensile samples showed such intergranular fracture behavior. The effects of loading rate and precipitation are discussed.

Keywords: ultra-high strength steel; carbide; loading rate

# **INTRODUCTION**

In developing high strength steels, it has been found that the carbon content in the steel is very important to the steel strength and toughness. With increasing carbon, the strength and hardness increase, but the toughness decreases, and the ductile to brittle transition temperature increases [1]. In order to develop low alloy ultra-high strength steels, with reasonable toughness, a medium carbon content is usually used, in the range of 0.2-0.6 wt.%. Other alloying elements are added to such medium carbon steels, such

<sup>\*</sup> Corresponding author.

Present address: Institute of Metal Research, Chinese Academy of Sciences (IMR CAS), Shenyang, China. Tel.: +86 024 23971517; fax: +86 024 23971517

E-mail address: yyshan@imr.ac.cn

as Cr, Ni, Mn, Mo, and Al, with their total content lower than 5%. This can increase the hardenability during quenching and refine the grain size.

The microstructure of ultra-high strength low alloy steels is usually designed to be a mixed structure, consisting of martensite laths strengthened by dislocations, carbide precipitates, and a small amount of residual austenite. This is achieved by quenching and low temperature tempering heat treatments, to maintain some toughness [2]. The 4030 steel is a superior low-alloy ultra-high strength martensite steel. It can reach an ultimate tensile strength higher than 1800 MPa, after tempering at around 200°C, but its fracture toughness  $K_{IC}$  is only 57 MPa.m<sup>-1/2</sup>.

In general, ultra high strength low alloy steels have relatively low cost, and thus are widely used, for examples for airplane landing gears and specialist bearing parts. However, because of their high strength and relatively poor toughness, brittle fracture can be a problem. This paper investigates the brittleness [3,4,5], in a tempered ultra-high strength low alloy martensitic steel, and the cleavage fracture phenomenon. Discussions are also made in relation to carbide precipitation in the martensitic steel.

#### MATERIAL AND METHODS

The composition of the experimental 4340 steel is given in Table 1. The steel was melted in a vacuum induction-melting furnace and then forged in bars with diameter of 14 mm. Both P and S contents were strictly controlled to benefit the toughness.

С	Mn	Si	Cr	Mo	Ni	O	N	S	Р
0.47	0.97	0.32	1.08	0.27	1.69	0.0047	0.0034	0.0033	0.0018

Table 1 Chemical composition of the ultra-high strength 4340 steel, in wt.%

Before the machining of any testing specimen, steel samples cut from the bar were subject to austenitizing at 850°C for 30 min and quenching in oil. Some of them were then tempered at 200°C for 90 min, and the others were directly fine machined for testing. The heat treatment schedules of the 4340 steel are given in Table 2. The tensile specimens had a gauge diameter of 5 mm and gauge length of 25 mm. The Charpy V-notched samples had the dimension of 55 mm  $\times$  10 mm  $\times$  10 mm. Both tests were carried out at room temperature.

The microstructure of the 4340 steel was observed by using light microscope (LM) and transmission electron microscope (TEM). The fracture surfaces of the broken samples were observed under a scanning electron microscope (SEM).

# **RESULTS AND DISCUSSION**

#### **Microstructure and Tensile Properties**

Fig. 1 presents the typical LM microstructure of the 4340 steel after oil-quenching and low temperature tempering, showing a fully tempered martensite microstructure. The microstructure under TEM observation is shown in Fig. 2. It can be seen that the tempered martensite microstructure of the 4340 steel mainly consists of lath martensite and dispersed  $\varepsilon$ -carbides with a lamellar shape of about 0.1 µm in length.

The tensile test results of the 4340 steel after various heat treatments are given in Table 2. The strength was decreased and the ductility was improved after tempering.

Heat treatment	Yield stress (MPa)	Tensile strength (MPa)	Elongation (%)	Reduction of area (%)
850°C (0.5 h), oil-quenching	1767	2451	3.2	2.8
850°C (0.5 h), oil-quenching, 200°C tempering (1.5 h)	1686	1966	11.2	45.7

Table 2 Heat treatment schedules and tensile properties of 4340 steel

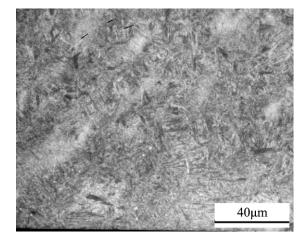


Fig. 1. LM microstructure of the 4340 steel oil-quenched from 850°C and tempered at 200°C.

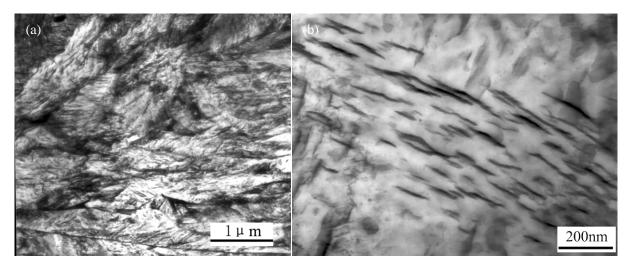


Fig. 2. TEM photographs of the 4340 steel oil-quenched from 850°C and tempered at 200°C. (a) Lath

martensite; (b) *ɛ*-carbides.

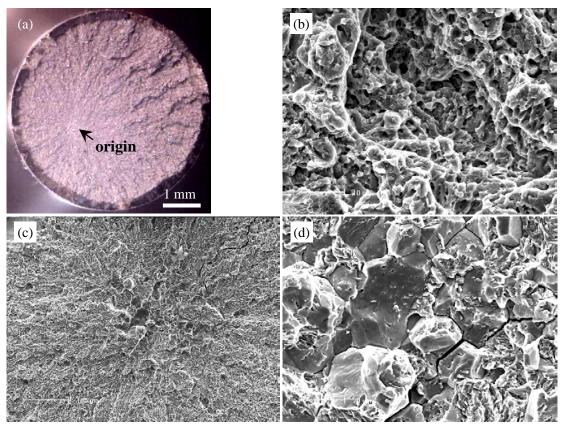


Fig. 3. Tensile fractography after 850°C/0.5 h, oil-quenching. (a) Macrograph of the fracture surface; (b) quasi-cleavage fracture; (c) origin of cleavage fracture; (d) intergranular crack at the origin.

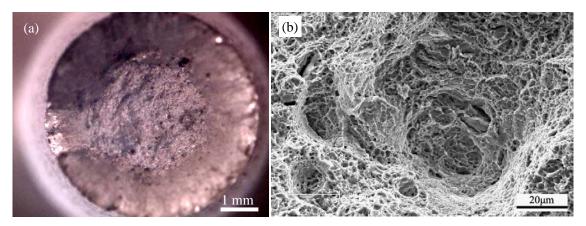


Fig. 4. Tensile fractography after 850°C/0.5 h, oil-quenching, 200°C tempering for 1.5 h. (a) Macroscopy of the fracture surface; (b) microscopy of dimple fracture.

## Fractography

The fracture surfaces of the broken samples were observed under SEM. The fracture surfaces of the tensile specimens of the steel after oil-quenching and oil-quenching followed by 200°C tempering are shown in Fig. 3 and Fig. 4, respectively. The fracture surface of the former sample exhibited remarkable quasi-cleavage fracture behavior. Moreover, when the cleavage rivers were traced back to the fracture origin, as shown in Fig. 3(a), it was found that there were intergranular cracks (Fig. 3(d)). Tracing back river patterns and fracture surface etching have shown new initiation mechanisms [6]. Quite normal cup-like shape fracture was observed on the surfaces of the tempered samples (Fig. 4). In many other steels, the center of the fracture surface was mainly characterized by the cup-like dimpled rupture [7].

Fig. 5 shows the fracture surfaces of the Charpy V-notched impact samples of the steel. The fracture surface of oil-quenching sample (Fig. 5(a)) exhibited a cleavage feature rather than the intergranular fracture observed on the tensile samples. Quasi-cleavage feature was observed on surfaces of the 200°C tempered sample, as shown in Fig. 5(b). These may be compared with typical fractograph taken from the fractured surface of a Charpy impact specimen, of a V-added AISI 4335 steel, tempered at 480°C [8].

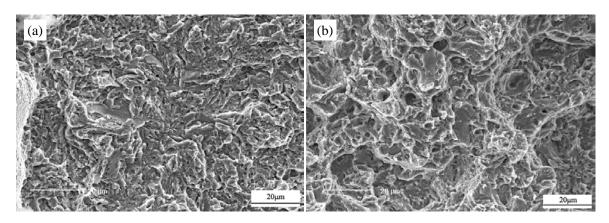


Fig. 5. Fractography of impact test samples. (a) Oil-quenched sample with cleavage fracture; (b) oil-quenched and 200°C tempered sample with quasi-cleavage feature.

# **Oil Quenching and Tempering**

As shown in Fig. 3(d), there is obvious intergranular fracture at the origin on the fracture surface of the quenched samples. Consequently, the ultra-high strength 4340 martensitic steel quenched without tempering is brittle. It is well known that martensite is a highly internally stressed microstructure [9] due to the excess carbon trapped interstitially during quenching.

However, when the samples were tempered even just at a low temperature of 200°C, the ductility could be completely changed. No brittleness occurred to sample at first subject to oil-quenching and then tempered at 200°C, as shown in Fig. 4. This means that, if the samples were tempered even just at as low a temperature as 200°C, the ductility will be completely changed. It is well known that many factors can affect the ductility regardless of chemical composition.

There was a very significant change in microstructure after tempering, carbide precipitation [10]. During the tempering, carbides, mainly  $\varepsilon$ -Fe<sub>3</sub>C, would precipitate, as shown in Fig. 2. Such precipitation was discussed by Wang et al. [11] and Lee et al. [12]. It is clearly shown that the worm-like  $\varepsilon$ -carbides are about 100 nm long and 10 nm wide.

#### Effect of Loading Rate and Microscopic Notch

No sign of intergranular fracture was observed on the surface of either the oil-quenched or the as-tempered Charpy impact samples, as shown in Fig. 5. However, for the tensile test, the loading rate was relatively low. Brittle fracture was observed in the origin of the fracture surface, with the characteristics of intergranular fracture, as shown in Fig. 3(d).

We suspected the pre-existence of microcracks after quenching. However, if there were microcracks, the strength could not be beyond 1500 MPa. In addition, if there were microcracks, the fracture origin should be derived from them, which should show us some previous crack characteristics at the origin. Generally, the microcracks due to quenching should be at the surface of the tensile samples, so the fracture origin should be at the surface. However, in our results, the origin is at the subsurface or at the corn. Lastly, if there truly were some cracks, they should still be there after tempering.

#### CONCLUSIONS

In summary, the brittle fracture behavior of a low-alloy ultra-high strength 4340 martensite steel was investigated in the present work and the following conclusions could be reached.

- The fracture surface of the oil-quenched tensile samples shows characteristic of intergranular fracture at the fracture origin.
- (2) Brittle fracture could be completely avoided by tempering even at as low a temperature as 200°C for a short time as 1.5 h. The ε-carbides are precipitated.
- (3) Loading rate plays a significant role. A lower loading rate such as tensile test than a higher loading rate such as impact test facilitates the intergranular fracture.

#### Acknowledgements

This work was financially supported by the National Key Technology R&D Program of China (Grant No. 2011BAE25B03) and the National Funds for Young Scholars of China (Grant No. 51201160). The authors thank Prof. Wei Yan at the Institute of Metal Research, Chinese Academy of Sciences for his valuable suggestions and discussions.

#### References

- Pickering, F. B. High-strength, low-alloy. In: Microalloying 75: Proceedings of an International Symposium on High-strength, Low-alloy Steels, October 1-3, 1975, Washington, DC, USA. Korchysky, M. (ed.), Union Carbide Corporation, New York, pp 9-31, 1977.
- Krauss, G. Heat treated martensitic steels: microstructural systems for advanced manufacture. ISIJ Int. 35:349-359, 1995.

- Horn, R. M., Ritchie, R. O. Mechanisms of tempered martensite embrittlement in low alloy steels. Metall. Trans. A 9:1039-1053, 1978.
- 4. Dieter, G. E. Mechanical Metallurgy. McGraw-Hill Book Co., New York, p 386, 1961.
- Kula, E. B., Anctil, A. A. Temperted martensite embrittlement and fracture toughness in SAE 4340 steel. J. Mater. ASTM 4:817-841, 1969.
- Echeverría-Zubiría, A., Linaza, M. A., Rodriguez-Ibabe, J. M. Influence of microstructure on cleavage fracture initiation micromechanisms in steels. In: ECF13: European Conference on Fracture: Fracture Mechanics: Applications and Challenges, September 6-9, 2000, San Sebastián.
- Farabi, N. et al. Microstructure and mechanical properties of laser welded DP600 steel joints. Mater. Sci. Eng. A 527:1215-1222, 2010.
- Jha, R. N. Dutta, K., Ray K. K. Effect of tempering on mechanical properties of V-added AISI 4335 steel. ISIJ Int. 50:607-612, 2010.
- 9. Ramírez, E. et al. Effect of microstructure on the sulphide stress cracking susceptibility of a high strength pipeline steel. Corros. Sci. 50:3534-3541, 2008.
- Mesquita, R. A. et al. Effect of silicon on carbide precipitation after tempering of H11 hot work steels. Metall. Mater. Trans. A 42:461-472, 2011.
- 11. Wang, X. D. et al. Carbide characterization in a Nb-microalloyed advanced ultrahigh strength steel after quenching-partitioning-tempering process. Mater. Sci. Eng. A 527:3373-3378, 2010.
- 12. Lee, J. B. et al. Kinetics of carbide formation for quenching and tempering steels during high-frequency induction heat treatment. Mater. Chem. Phys. 129:365-370, 2011.