Haitao Liu*, Weiqing Chen, Fengyou Qi and Wenying Li

Effect of Rare Earth Yttrium on the Hot Ductility of Eco-friendly Bi-S Based Free Cutting Steel

Abstract: The hot ductility of eco-friendly Bi-S based free cutting steels micro-alloyed with and without yttrium was studied using a Gleeble-1500 thermal–mechanical simulator over the temperature range 850–1200 °C. The results showed that the addition of rare earth yttrium had a substantial improvement in the hot ductility of Bi-S based free-cutting steel, especially at 1000 °C. The beneficial effect of yttrium on the hot ductility of Bi-S based free-cutting steel at the temperature no less than 1000 °C was mainly associated to the refinement of austenite grain size, which could effectively reduce the segregation density of bismuth at the grain boundary, and the lowering of the DRX onset temperature by yttrium addition. At 850–950 °C, the improvement of the hot ductility in these steels by yttrium addition might be attributed to the reduction of the low melting point sulphides at grain boundary and the refinement of the austenite grain size. However, the hot ductility of these steels micro-alloyed with yttrium was still poor at 850–950 °C, which was mainly owning to the presence of pro-eutectoid ferrite films and the absence of dynamic recrystallization as well as the segregation of liquid bismuth films at austenite grain boundaries.

Keywords: free-cutting steel, yttrium, hot ductility, liquid bismuth film, intergranular embrittlement

PACS® (2010). 81.40.-z

***Corresponding author: Haitao Liu:** State Key Laboratory of Advanced Metallurgy, and School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, China. E-mail: liuhaitao1204@126.com
Weiqing Chen: State Key Laboratory of Advanced Metallurgy, and School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, China
Fengyou Qi, Wenying Li: Qingdao Iron and Steel Group Co., Ltd, Qingdao 266043, China

1 Introduction

Pb-S based free cutting steel has been widely used in the manufacture of machined parts because they provide good machinability, and thus good productivity [1–2]. But it is well-known that a considerable amount of lead in the steel will generate lead fumes during the machining and recycling processes, which is harmful to humans. So the need for the replacement of Pb-S based steels has been strongly suggested, and an eco-friendly Bi-S based free cutting steel has been developed, in which lead is replaced by bismuth [1–2]. The Bi-S based steels have a similar excellent machinability compared to the Pb-S based steels since bismuth and lead have similar physical properties [3–6].

Unfortunately, the eco-friendly Bi-S based free cutting steels are not yet widely used for commercial purposes because hot rolling is more difficult than that of the Pb-S based free cutting steels [7–12]. These steel easily crack during hot rolling and have a poor ductility [9–12]. In addition, recent investigations on these steels were mainly focused on their machinability rather than their hot ductility [3–6, 10]. However, some research has been suggested that rare earth element could segregate to the grain boundaries preferentially and suppress the segregation of impurity effectively when the rare earth and impurity elements existed simultaneously in the steel, then increasing the grain boundary cohesion strongly and in turn improving the steel hot ductility [13–16]. But further studies about the effect of rare earth on the hot ductility of steel are still necessary. In order to find an available way to improve the hot ductility of the Bi-S based free cutting steel, the addition of rare earth yttrium (Y) to the steel has been considered here. The hot ductility of eco-friendly Bi-S based free cutting steel with Y addition in the temperature range of 850–1200 °C has been reported in this article, and the manner in which a Y addition affects the hot ductility has also been investigated in relation to the microstructural changes.

2 Experimental procedures

The tested steels with and without Y were melted in a vacuum induction furnace at a temperature of 1600 °C and were cast to 6.5 kg ingots. The chemical compositions are shown in Table 1. These ingots were first reheated at 1200 °C for 2 h in a heating furnace, and then, an air

Table 1: Chemical compositions of tested steels, mass%

Steels	C	Si	Mn	P	S	O	N	Bi	Y
1	0.07	0.009	1.25	0.050	0.31	0.0228	0.0042	0.17	–
2	0.08	0.03	1.42	0.057	0.30	0.0107	0.0056	0.16	0.0064
3	0.07	0.016	1.35	0.049	0.33	0.0140	0.0044	0.16	0.021

forging hammer was used to forge them into wire rods with a diameter of 15 mm. During the forging process, the temperature was always >1050 °C, and finally, the wire rods were air cooled to room temperature. Specimens with dimensions of 10 mm diameter and 120 mm length for hot tensile testing were prepared from the wire rods.

The hot tensile test was performed with a Gleeble-1500 thermal-mechanical simulator under argon protection atmosphere using a constant strain rate of 10^{-2} s^{-1} in the temperature range of 850–1200 °C. The specimens were first heated with a heating rate of 10 °C s^{-1} to 1350 °C and held there for 5 min, and then cooled with a cooling rate of 3 °C s^{-1} to each testing temperatures with an interval of 50 °C, at which they were maintained for 1 min before tensile testing. After rupture, the samples were immediately quenched by water spraying to maintain the microstructure at the testing temperature, and the reduction in area R_A was measured to evaluate their hot ductility.

The fracture surfaces of the tensile specimens were examined using scanning electron microscopy (SEM). The longitudinal sections close to the point of fracture were prepared and etched with a picral solution, and then, the microstructures and the inclusions were detected by a combination of the SEM in backscattered electron (BSE) mode and secondary electron (SEI) mode, and the energy dispersive X-ray spectroscopy (EDS).

3 Experimental results

3.1 Hot ductility of steels with and without Y

The R_A of all tested steels with and without Y addition in the testing temperature range is shown in Fig. 1. It can be seen that Y has a beneficial effect on the hot ductility. The addition of Y could increase the hot ductility of the Bi-S based free cutting steels over the temperature range 850–1100 °C, especially at 1000 °C, and there was little difference in the hot ductility of these steels when the Y content was 0.0064% and 0.021% respectively.

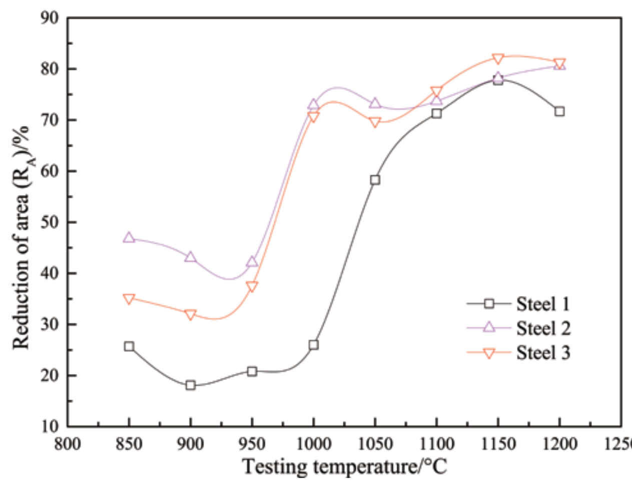

Fig. 1: Hot ductility curves of all tested steels

The hot ductility of Bi-S based free cutting steels with Y was all relatively good when the temperature was no less than 1000 °C, and the R_A was >70%, which illustrated that the steels with Y could be suitable for rolling provided the finish rolling temperature did not fall <1000 °C. However, the hot ductility of the steels with Y was still very poor in the temperature range 850–950 °C.

3.2 Fracture morphology of the steels

The fracture surfaces of the tensile tested specimens were observed by SEM. The steels with and without Y both exhibited ductile dimple fractures when the hot ductility was good, but intergranular brittle fractures when the hot ductility was poor. The typical SEM photos of these fractures were shown in Fig. 2. At 900 °C, the fractures of steel 1 and steel 2 both exhibited intergranular brittle failure (Figs. 2a and b), which agrees with the low ductility values given in Fig. 1.

Steel 2 with Y addition at 1000 °C showed a ductile failure (Fig. 2d), but the fracture of steel 1 without Y addition was mainly intergranular (Fig. 2c), which could indicate that the grain boundary strength of steel 1 without Y was lower than that of the steel 2.

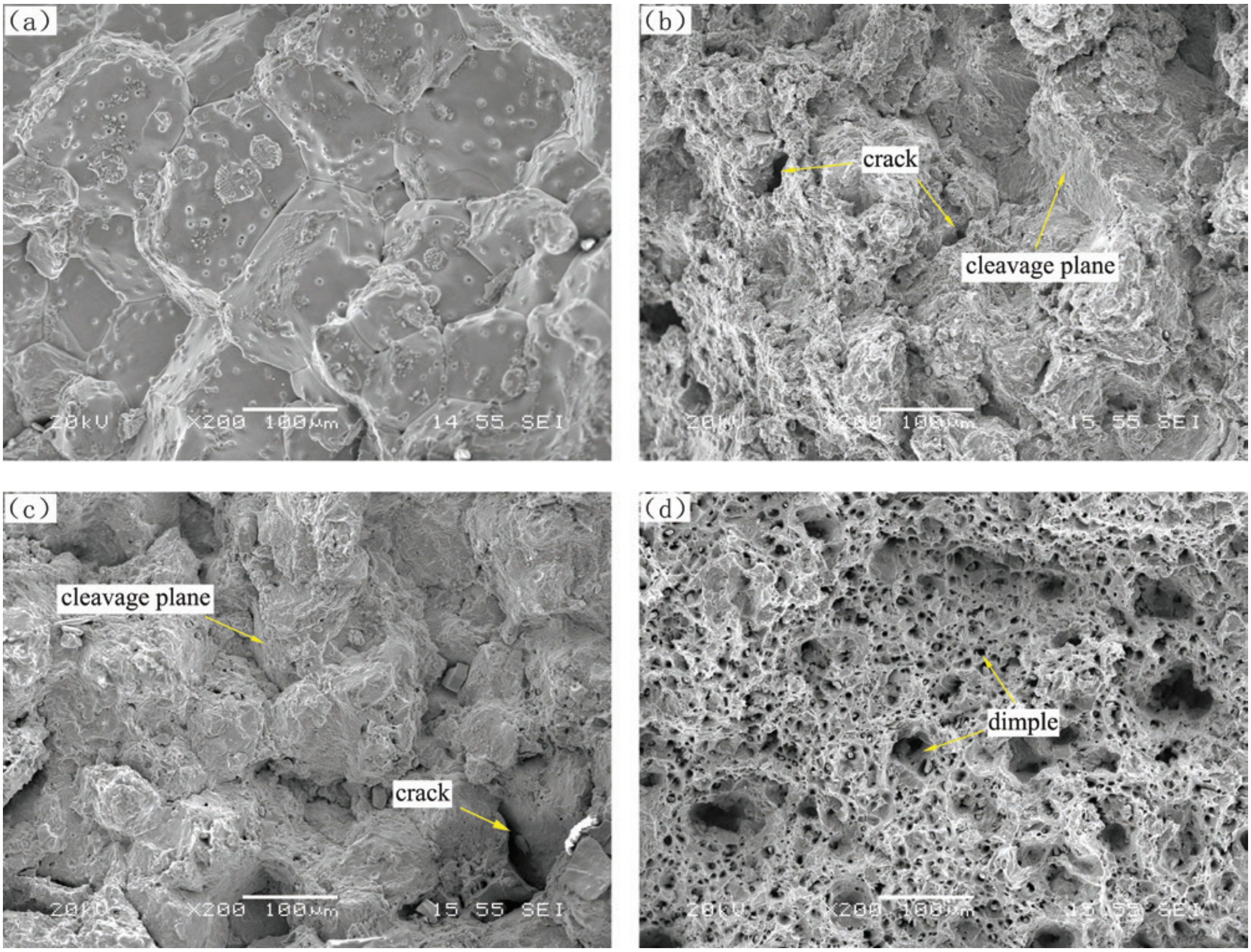

Fig. 2: Fracture morphology of tensile tested samples: (a) steel 1 at 900 °C; (b) steel 2 at 900 °C; (c) steel 1 at 1000 °C; (d) steel 2 at 1000 °C

4 Discussion and analysis

4.1 Effect of Y on bismuth grain boundary segregation

From the observation and analysis of the longitudinal microstructures of all quenched fractures in austenite single-phase region using the SEM in SEI and BSE modes, it has been found that the rare earth Y could suppress the segregation of bismuth to some extent at grain boundaries in Bi-S based free cutting steel. Steel 1 without Y at 950, 1000 and 1050 °C, but steel 2 with Y only at 950 °C, showed cracks along austenite grain boundaries accompanied by the presence of bismuth films at the boundaries. The typical longitudinal microstructures of the quenched fracture are shown in Fig. 3, and the EDS spectra and linescan of the liquid bismuth films are shown in Fig. 4.

According to Chang et al. there is a negligible solid solubility of bismuth in solid steel and the solubility of bismuth decrease with decreasing temperature in γ-iron, then the lower the temperature, the more bismuth single substance phase will be precipitated in the solid steel [16–18]. Therefore, although steel 2 was added with rare earth Y, there was still some bismuth films precipitated along the grain boundaries at 950 °C. Meanwhile, Pure Bi has a melting point of 271.3 °C and so will exist in liquid form at the tensile testing temperatures and cause liquid metal embrittlement [19–21]. If a crack forms on deformation at the testing temperature, the liquid bismuth in the steel would flow into the crack tip so reducing the binding energy, making it easier to nucleate and propagate cracks, particularly at the austenite grain boundaries [19–21]. Then the bismuth films present at austenite grain boundary would significantly reduce the strength of the grain boundary and cause grain boundary embrittlement.

Meanwhile, a comparison of the typical metallographs of the steels with and without Y at 1050 °C was shown in Fig. 5. It can be seen that the addition of Y has an

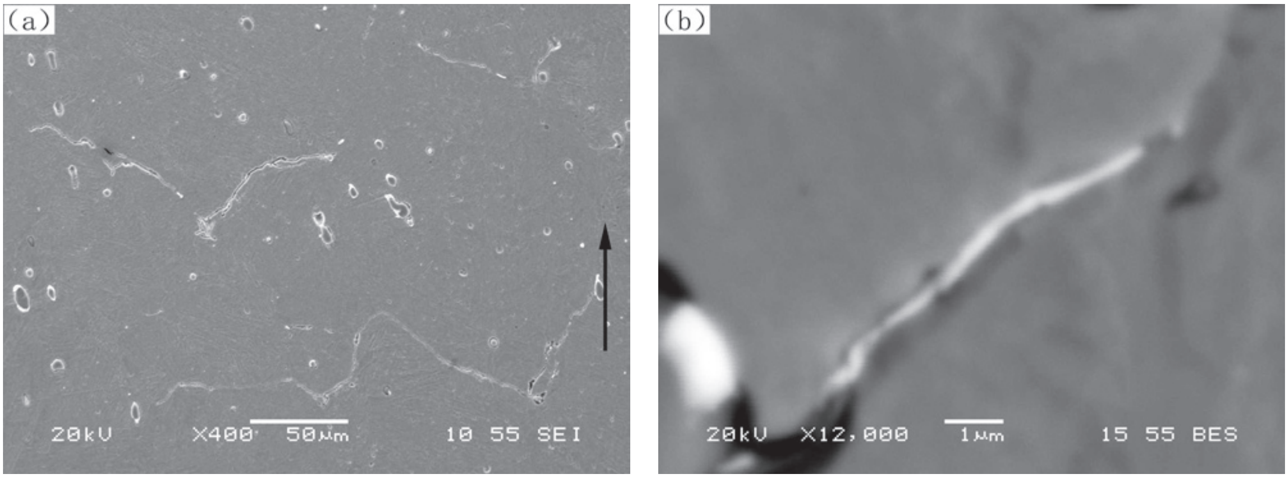

Fig. 3: Typical longitudinal microstructures of quenched fractures at 950–1050 °C: (a) morphology of cracks along austenite grain boundary; (b) segregation of liquid bismuth film. (The arrow in (a) indicates the tensile direction.)

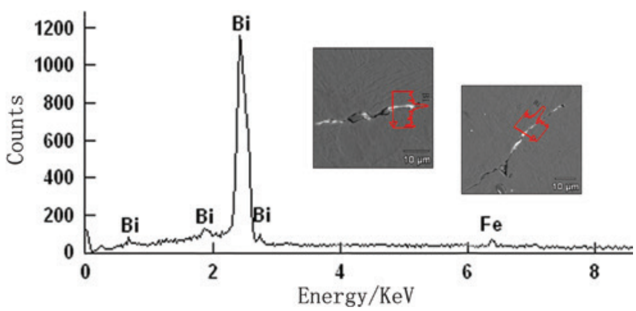

Fig. 4: Spectra (EDS) and linescan of liquid bismuth films

effect of macrostructure refinement on the steel, the austenite grain size of the steel was refined obviously, which might be attributed to the formation of rare earth oxysulfide particles with high-melting point and low lattice misfit with matrix, or/and the occurrence of the dynamic recrystallization (Fig. 6). As we know, the finer the grain size, the grater the grain boundary area. With the increase of the grain boundary area, the density of bismuth segregating at the grain boundary could be reduced, so the grain boundary was purified and strengthened. Therefore, the beneficial effect of yttrium on the hot ductility of Bi-S based free-cutting steel at the temperature no less than 1000 °C would be mainly associated to the refinement of austenite grain size which could effectively reduce the segregation density of bismuth at the grain boundary.

4.2 Effect of Y on dynamic recrystallization

The flow stress curves for steel 1–3 are shown in Fig. 6. The temperature for the onset of dynamic recrystallization (DRX) can generally be obtained from the flow stress

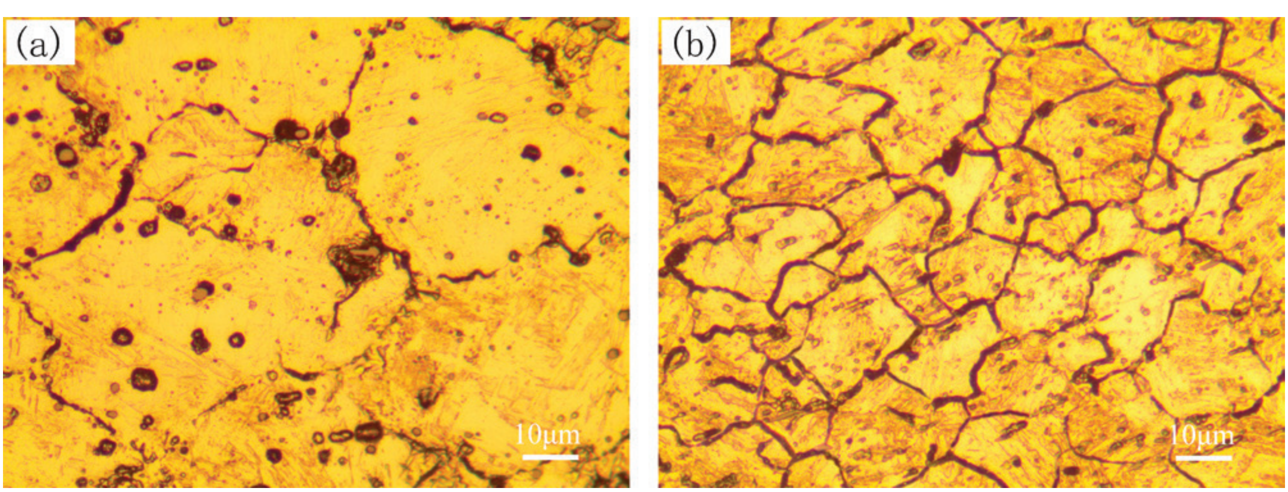

Fig. 5: A comparison of the typical metallographs of the steels at 1050 °C: (a) steel 1; (b) steel 2

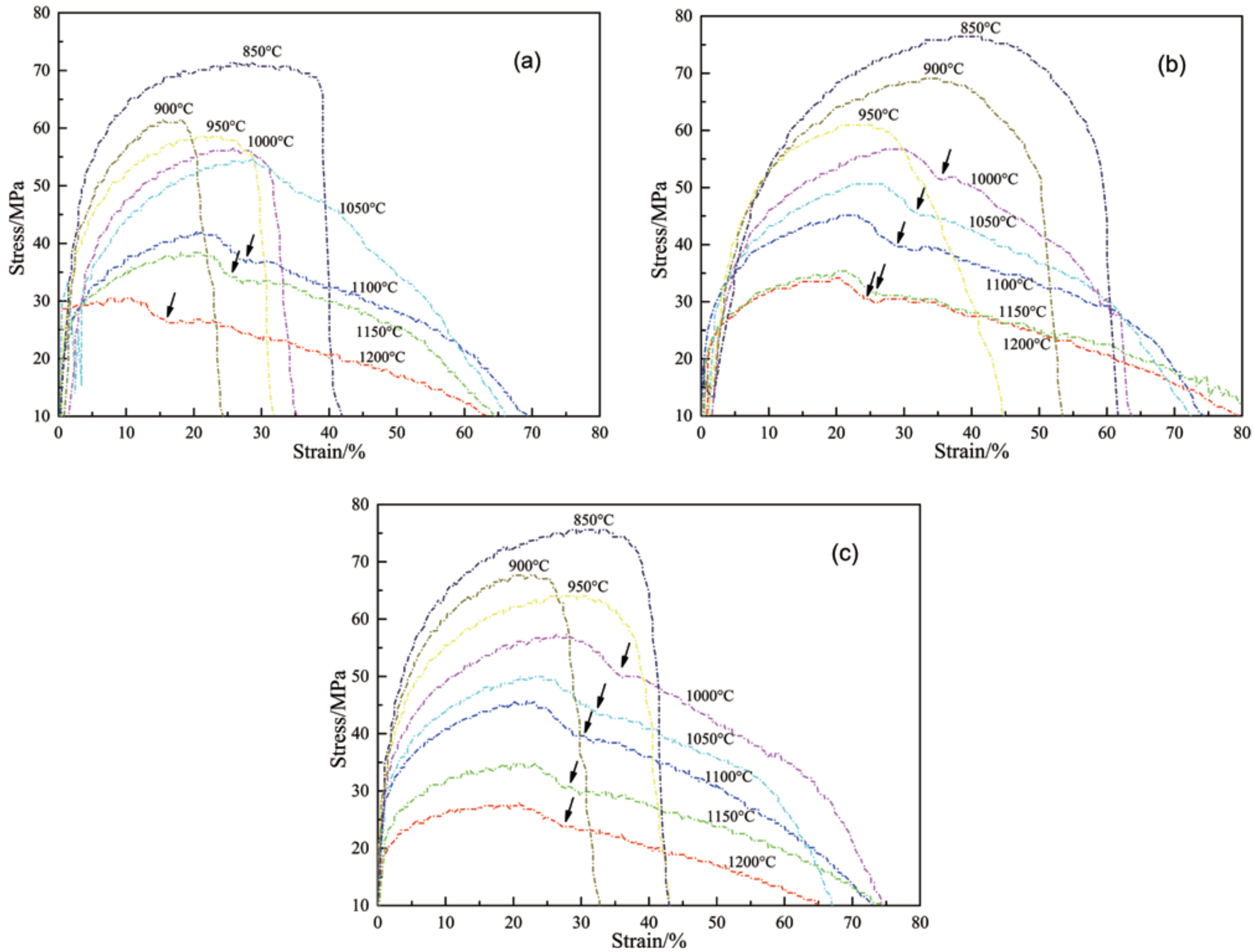

Fig. 6: Flow stress-strain curves as function of tensile temperature for examined steels (arrows indicate DRX): (a) steel 1; (b) steel 2; (c) steel 3

curves by noting the first test temperature at which load fluctuations occur in the austenite, because the occurrence of DRX will provide an additional softening to that provided solely by dynamic recovery to offset part of the work hardening caused by deformation and in turn the flow stress passes will fluctuate in a single peak or a multiple peak type [22–24]. Mintz and Mohamed have reported that DRX is essential for good ductility, and ductility in high S steel improves only at higher temperatures when DRX is well established [23–26]. From Fig. 6, it can be seen that the onset temperature of DRX for the steels with and without Y was 1100 and 1000 °C respectively. The addition of Y had a remarkable effect on lowering the onset of DRX for 100 °C, and the recovery temperature of hot ductility for Bi-S based free cutting steels agreed with the onset temperature of DRX. DRX, i.e. grain boundary migration, can move grain boundaries away from microcracks and prevent the coalescence and growth of the voids at grain boundaries, then improving the hot ductility [22, 23, 27–31]. Fig. 7 was the evidence of DRX in steel 2 with Y at 1000 °C.

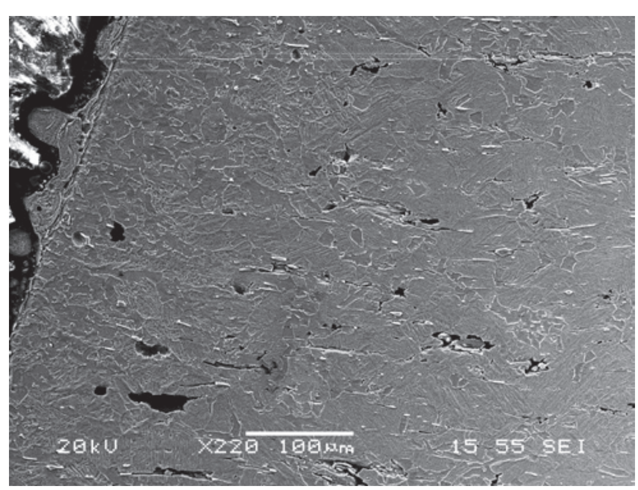

Fig. 7: Evidence of DRX in steel 2 with Y at 1000 °C (SEM image)

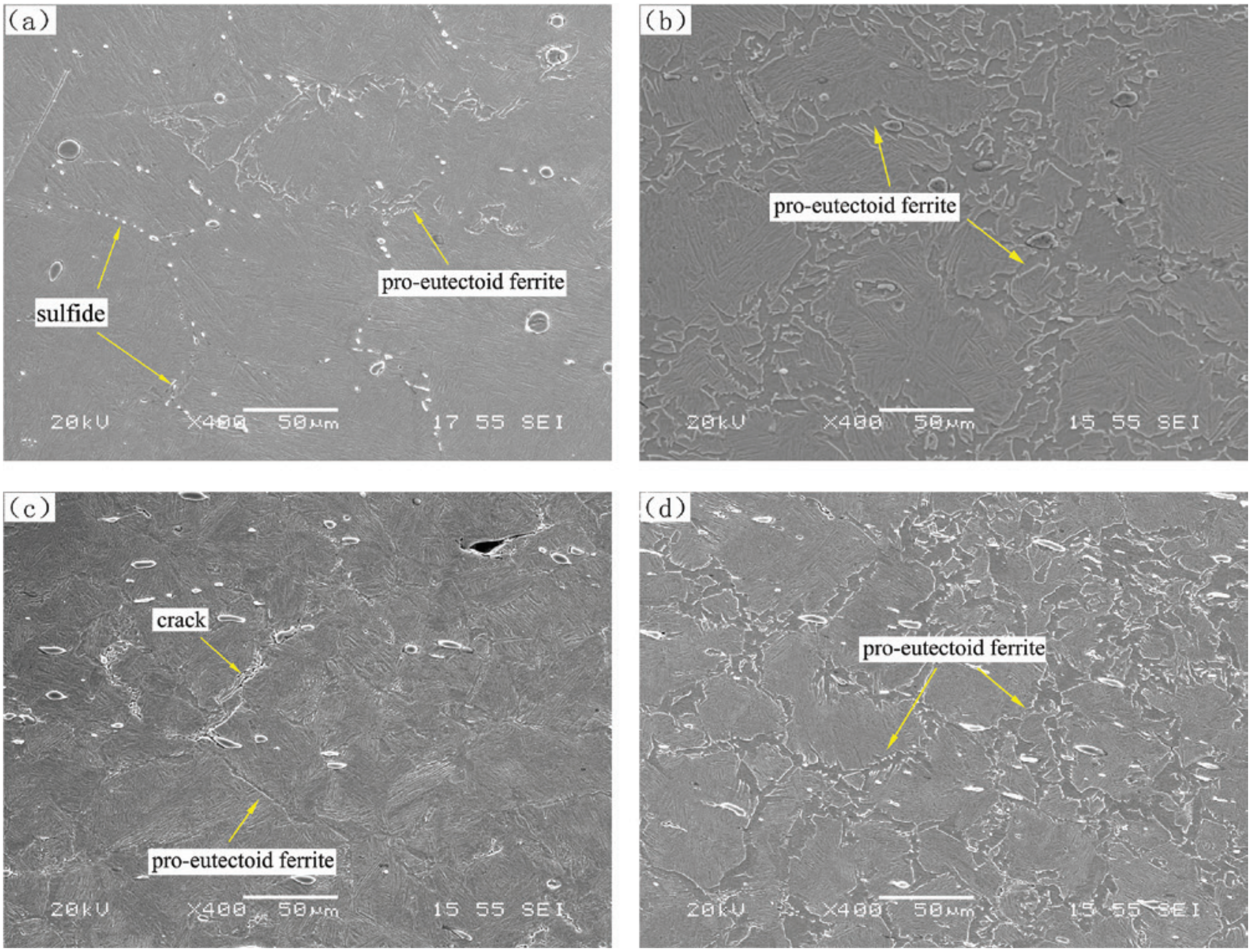

Fig. 8: Typical longitudinal section microstructures near quenched fractures for both Y free and containing steels: (a) steel 1 at 900 °C; (b) steel 1 at 850 °C; (c) steel 2 at 900 °C; (d) steel 2 at 850 °C

4.3 Microstructure in austenite/ferrite two phases region

The typical longitudinal microstructures of the quenched fractures in austenite/ferrite two phases region were also observed by using SEM, and the results were shown in Fig. 8. It can be seen as follows:

The proeutectoid ferrite films were formed along the grain boundary in both Y containing and Y free steels at 850–900 °C, while some bismuth films still existed at the grain boundary. The strength of these proeutectoid ferrite was lower than that of austenite, then grain boundary sliding and cracks along the ferrite film would be caused by strain concentration during the deformation process [30–33]. From Fig. 6, it also can be seen that the DRX in these steels did not occur in this temperature range, which would lead to the poor ductility. However, the precipitation of low melting point sulphides, which were harmful to hot ductility, could be reduced by the addition of rare earth Y, because the rare earth Y could capture part of sulfur in the grains and reduce the segregation of some additional dissolved sulfur at the grain boundaries. From Figs. 8a and c, it can be seen that some Fe rich (Fe,Mn)S phases were not formed along the grain boundary in steel 2 but in steel 1. But the hot ductility of steel 2 was still relatively poor because of the formation of proeutectoid ferrite films along the grain boundary.

From Fig. 8, it also can be seen that the addition of Y in Bi-S based free cutting steel could refine the austenite grain size. The finer the grain size, the better the hot ductility, because finer grain was beneficial to restraint the sliding of grain boundary [34–35]. Then, the hot ductility of the Y containing steels was higher than that of the Y free steels in this temperature range even though the hot ductility of them was all relatively poor.

Therefore, the improvement of the hot ductility in the steels by Y addition at 850–900 °C might be attributed to the inhibitory effect of Y on sulfur segregation to grain boundary and the refinement of austenite grain size. The poor ductility of these steels was mainly owning to the presence of the ferrite films and the unrecrystallised austenite as well as the segregation of liquid bismuth films.

5 Conclusions

The addition of Y could obviously increase the hot ductility of Bi-S based free cutting steels over the temperature range 850–1100 °C, especially at 1000 °C.

The beneficial effect of yttrium on the hot ductility of Bi-S based free-cutting steel at the temperatures no less than 1000 °C was mainly associated to the refinement of austenite grain size, which could effectively reduce the segregation density of bismuth at the grain boundary, and the lowering of the DRX onset temperature. The reduction of the segregation of liquid bismuth films could increase the strength of the grain boundary. DRX could move grain boundaries away from microcracks and preventing their coalescence at grain boundaries.

At 850–950 °C, the improvement of the hot ductility in the steels by Y addition might be attributed to the reduction of the low melting point sulphides at grain boundary and the refinement of the austenite grain size. However, the hot ductility of these steels micro-alloyed with Y was still poor, which was mainly owning to the presence of pro-eutectoid ferrite films and the absence of DRX as well as the segregation of liquid bismuth films at austenite grain boundaries.

Received: August 3, 2013. Accepted: October 31, 2013.

References

[1] K. Sander, J. Lohse and U. Pirntke, *Heavy Metals in Vehicles – Final Report*, Institut für Ökologie und Politik GmbH, Hamburg, (2000), pp. 4–44.

[2] J. Lohse, K. Sander and M. Wirts, *Heavy Metals in Vehicles II – Final Report*, Institut für Ökologie und Politik GmbH, Hamburg, (2001), pp. 45–91.

[3] R. Philip, B. Volker and E. Ingo, *Steel Res. Int.*, **178** (12) (2007) 908–914.

[4] M. Somekawa, M. Kaiso and Y. Matsushima, *Kobelco Technology Review*, (1) (2001) 9–13.

[5] L. Vea, *SAE Technical Paper on 'New steel bar products and processing for automotive applications'*, Society of Automotive Engineers, Detroit, (1997), pp. 77–82.

[6] H.T. Liu and W.Q. Chen, *Steel Res. Int.*, **83** (12) (2012) 1172–1179.

[7] J. Klapsia, *Hutnicke Listy*, (9–11) (2003) 18–19.

[8] J. Klapsia and E. Karczmarczyk, *Hutnicke Listy*, (10–12) (2000) 36–38.

[9] Y. Kim, H. Kim, S.Y. Shin, K. Rhee, S.B. Ahn, D.L. Lee, N.J. Kim and S. Lee, *Metall. Mater. Trans.*, **42A** (2011) 3095–3105.

[10] S.K. Cho, *J. Korean Inst. Met. Ma.*, **34** (7) (1996) 822–829.

[11] H. Kim, M. Kang, S.Y. Shin and S. Lee, *Mat. Sci. Eng. A*, **568** (2013) 8–19.

[12] Y. Kim, H. Kim, S.Y. Shin, K. Rhee, S.B. Ahn, D.L. Lee, N.J. Kim and S. Lee, *Metall. Mater. Trans.*, **43A** (2012) 882–892.

[13] P.E. Waudby, *Int. Mater. Rev.*, **23** (2) (1978) 74–98.

[14] W.G. Wilson, L.J. Heaslip and I.D. Sommerville, *JOM*, **37** (9) (1985) 36–41.

[15] L. Chen, X.C. Ma, L.M. Wang and X.N. Ye, *Mater. Design*, **32** (4) (2011) 2206–2212.

[16] L. Chang and M.E. Schlesinger, *Metall. Mater. Trans.*, **29B** (1998) 1371–1372.

[17] A.V. Ryabov, D.Y. Povolotskij and S. Archugov, *Chernaya Metallurgiya*, (7) (2001) 3–6.

[18] A.Y. Zaslavskij, Y.A. Ageev, N.N. Kuz'kina, S.N. Kovaleva, S.M. Lukavyj and S.V. Kuznetsov, *Metally*, (2) (2002) 58–64.

[19] H.J. McQueen, S. Yue, N.D. Ryan and E. Fry, *J. Mater. Process. Tech.*, **53** (1995) 293–310.

[20] G. Bernsmann, M. Bleymehl, R. Ehl and A. Hassler, *Stahl Eisen*, **121** (2) (2001) 87–91.

[21] J. Klapsia and E. Karczmarczyk, *Hutnicke Listy*, (10–12) (2000) 36–38.

[22] E. López-Chipres, C. Maldonado, A. Bedolla-Jacuinde, M. El-Wahabi and J.M. Cabrera, *Mat. Sci. Eng. A*, **480** (1–2) (2008) 49–55.

[23] B. Mintz, R. Abushosha and J.J. Jonas, *ISIJ Int.*, **32** (2) (1992) 241–249.

[24] B. Mintz and Z. Mohamed, *Mater. Sci. Technol.*, **4** (10) (1988) 895–902.

[25] A. Cowley and B. Mintz, *Mater. Sci. Technol.*, **20** (11) (2004) 1431–1439.

[26] B. Mintz, R. Abu-Shosha and M. Shaker, *Mater. Sci. Technol.*, **9** (10) (1993) 907–914.

[27] I. Mejía, A. Bedolla-Jacuinde, C. Maldonado and J.M. Cabrera, *Mat. Sci. Eng. A*, **528** (13) (2011) 4468–4474.

[28] L.H. Chown and L.A. Cornish, *Mat. Sci. Eng. A.*, **494** (1) (2008) 263–275.

[29] F. Zarandi and S. Yue, *Metall. Mater. Trans.*, **37A** (7) (2006) 2316–2320.

[30] S.H. Song, A.M. Guo, D.D. Shen, Z.X. Yuan, J. Liu and T.D. Xu, *Mat. Sci. Eng. A*, **360** (1) (2003) 96–100.

[31] E. López-Chipres, C. Maldonado, A. Bedolla-Jacuinde and J.M. Cabrera, *Mat. Sci. Eng. A*, **460–461** (2007) 464–470.

[32] C. Zhang, E.B. Yue and S.T. Qiu, *Continuous Casting*, (6) (2008) 6–10.

[33] K.C. Cho, D.J. Mun, Y.M. Koo and J.S. Lee, *Mat. Sci. Eng. A*, **528** (10) (2011) 3556–3561.

[34] H. Zhang, W.F. Cui, J.J. Wang and C.M. Liu, *Trans. Mater. Heat Treat.*, **32** (1) (2011) 61–66.

[35] Q. Lin, F. Guo and X.Y. Zhu, *J. Chin. Rare Earth Soc.*, **24** (6) (2006) 729–733.