


Effects of Nb and V microalloying on the thermoplasticity of new martensitic low-density steels

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ABSTRACT

By performing tensile tests in the temperature range of 800°C to 1200°C, the thermoplastic behavior of microalloyed and unmicroalloyed new martensitic low-density steels were investigated, and the mechanism of the effect of Nb and V microalloying on the thermoplasticity was revealed. The results showed that both microalloyed and unmicroalloyed steels have good thermoplasticity and the plasticity increased with increasing deformation temperature. The microalloyed steels above 1000°C could have their high-temperature plasticity significantly enhanced by Nb, V microalloying, while the microalloyed steels at or below 1000°C could have their plasticity reduced. When the deformation temperature exceeds 1000°C, complete recrystallization occurs in both microalloyed and unmicroalloyed steels. The Nb, V microalloys were able to refine the recrystallized grains, which could obtain a stronger resistance to crack expansion and give the microalloyed steels better high-temperature plasticity. When the deformation temperature at or below 1000°C, the unmicroalloyed steel exhibited significant recrystallization. The presence of numerous small-sized NbC precipitation phases, abundant in the microalloyed steel, hindered the recrystallization. This made dynamic recrystallization of microalloyed steels almost non-existent when deformation occurred at lower temperatures, which lead to lower plasticity compared to the unmicroalloyed steel.

Keywords: Low-density steel; Microalloying; Mechanical properties; Thermoplasticity.

1. INTRODUCTION

The automotive industry has been driving rapid economic development, but at the same time has brought enormous pressure on the environment and energy [1]. How to reduce the energy consumption of automobiles has become the focus of research in the automotive field nowadays. Fe-Mn-Al-C low-density steel has both low density and high strength, which makes it one of the key research directions for automotive steel in the new era [2, 3]. It has been shown that due to the relatively light molecular mass of Al element, it can effectively reduce the density of steel. It also inhibits the expansion of the austenitic phase zone and raises the temperature and work-hardening rate of the two-phase zone, thus improving the plasticity of the steel [4]. At present, countries mainly design low-density steels for hot-rolled sheets for automobile bodies. Steel types are mainly ferritic, austenitic or ferrite-austenitic duplex type, while other matrix types of low-density steels of other matrix types are rarely studied.

Microalloying can improve steel properties, by adding V, Nb and Cr, the steel is simultaneously hard and tough by triggering solid solution strengthening and precipitation strengthening [5, 6]. Previous research shows that the addition of Nb elements to N-free steels tends to produce small-sized NbC precipitation phases of tens to hundreds of nanometers [7, 8]. Previous research shows that microalloying elements such as Nb and V have a significant effect on the recrystallization behavior of steel, which is mainly manifested by delaying the onset of dynamic recrystallization and refining the recrystallized grains [9, 10]. DU *et al.* [11] find that the thermoplasticity of the material increases with the degree of recrystallization during high-temperature deformation by studying the tensile properties of NiFeCoCrMn high-entropy alloy after machining. CUI *et al.* [12] in their study of the effect of Al content in Al-TRIP steels on their high-temperature mechanical properties find that recrystallization to form uniform grains can disperse deformation and reduce the strain difference between intercrystalline and grain boundaries, which further reduces stress concentration to enhance the plasticity of the steel. By investigating the thermoplastic properties of niobium microalloying with niobium containing 1800

MPa grade at different high-temperature conditions and analyzing the fracture failure mechanism of 0.04Nb steel in different temperature intervals, LIU *et al.* [13] also shows that the precipitated phase of Nb inhibits dynamic recrystallization.

In this paper, we used the Gleeble-3800 thermal simulation tester to perform high-temperature tensile tests on unmicroalloyed and microalloyed martensitic low-density steels, respectively. Plotting its high-temperature thermoplastic curves and characterizing the fracture morphology and the metallographic organization near the fracture. Revealing the effect of Nb and V microalloying on the high-temperature plastic behavior of a new martensitic low-density steels. Explore the way for the production and application of new martensitic-type low-density steel in forging-type automotive parts, and provide new ideas for the lightweight of automobiles.

2. MATERIALS AND METHODS

This study used a vacuum induction furnace to smelt 1t ingots, produced as $\phi 130\text{mm}$ bars by forging and annealed at 700°C after cooling. The alloy compositions of the experimental steels determined by EDS spectroscopy were shown in Table 1. In this study, experimental steel A was a non-Nb, V microalloyed steel, and experimental steel B was an Nb, V microalloyed steel. Measured with the density measurement module of the sartorius BSA224S electronic analytical balance, the density of experimental steel A is 7.614g cm^{-3} , and the density of experimental steel B is 7.677g cm^{-3} , which is about 3% and 2.2% lower than conventional steel. The designed experimental steel can effectively reduce the density after adding aluminum and has the characteristics of lightweight.

The Gleeble-3800 thermal simulation machine was used to perform thermal simulation experiments on high-temperature tensile specimens of $\Phi 10\text{mm} \times 120\text{mm}$ in size. The thermomechanical process was shown in Figure 1. The experimental steels were heated at $10^\circ\text{C} \cdot \text{s}^{-1}$ to 1230°C for 3 min, and then cooled at $10^\circ\text{C} \cdot \text{s}^{-1}$ to 800°C , 850°C , 900°C , 950°C , 1000°C , 1050°C , 1100°C , 1150°C and 1200°C and held for 1 min, respectively. The tensile test was performed at $0.1/\text{s}$ under argon protection, and the fracture was cooled by water spray immediately after pulling. Used a wire cutter to take a 3 cm long specimen from the fracture and cut the specimen through the extended diameter. After grinding and polishing the cross section, etching was performed using a 4% mass fraction solution of ethanol nitrate to reveal the microstructure. Samples from different treatments were observed using a ZEISS Axioscope 5 optical microscope (OM) and a JEOL JSM-6390LV model scanning electron microscope (SEM). Nano Measurer 1.2 software was used to measure the grain size, and the grain size was statistically analyzed by Origin software.

3. RESULTS

3.1. Thermoplastic behavior

The stress-strain curves of experimental steels A and B at different deformation temperatures are shown in Figure 2. The maximum stress of experimental steels A and B decreases with the increase of the tensile temperature. The transient stress reduction could be observed in all the initial stages of experimental steel A, as

Table 1: Chemical composition of the experimental steel (mass fraction, %).

TYPE	C	Al	Mn	Si	P	S	Cr	V	Nb	Fe
steel A	0.44	3.62	5.14	0.45	0.025	0.003	0.11	–	–	Bal.
steel B	0.45	3.38	5.07	0.48	0.03	0.003	0.11	0.13	0.09	Bal.

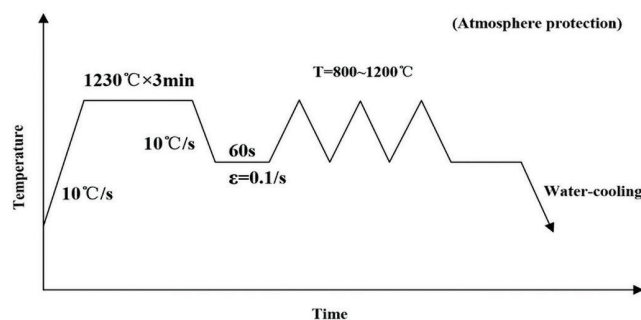


Figure 1: Thermoplastic experimental process scheme.

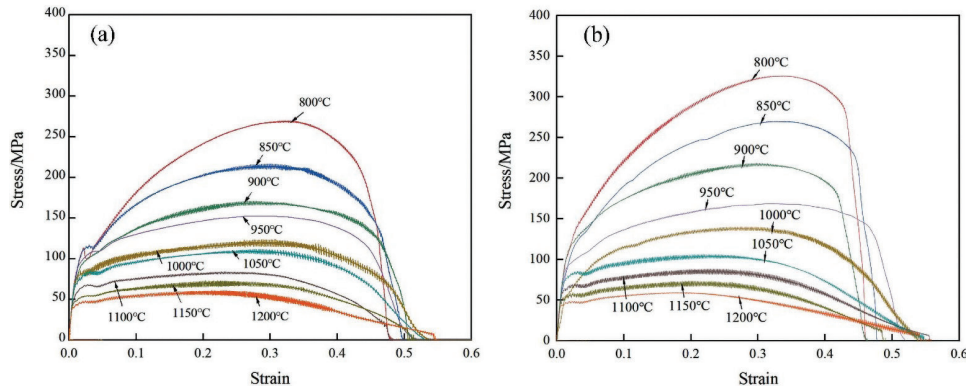


Figure 2: Stress-strain relationship curve (a) steel A; (b) steel B.

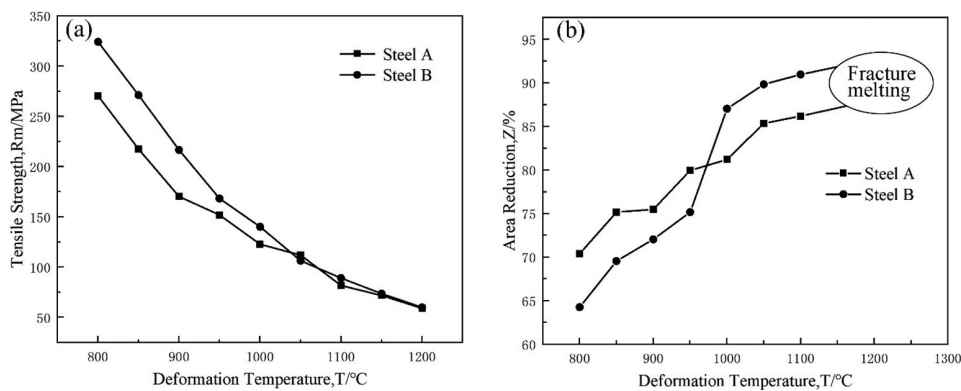


Figure 3: Mechanical properties (a) tensile strength; (b) area reduction.

shown in Figure 2(a). But the experimental steel B only showed a transient stress drop when the deformation temperature exceeded 1000°C, as shown in Figure 2(b).

The changes in mechanical properties of experimental steels A and B steel at different temperatures were shown in Figure 3. The tensile strength of experimental steels A and B gradually decreased with the increase of deformation temperature. At 800°C, the tensile strength of experimental steels A and B differed considerably, and the tensile strengths were equal after exceeding 1000°C, as shown in Figure 3(a). The sectional shrinkage of experimental steels A and B increased gradually with the increase of deformation temperature. The plasticity of experimental steel A was significantly lower than that of experimental steel B. Meanwhile, when the deformation temperature increased from 950°C to 1000°C, the section shrinkage of experimental steel B increases significantly, as shown in Figure 3(b).

3.2. Microstructural features

The SEM photographs of the tensile fractures of experimental steels A and B at different deformation temperatures were shown in Figures 4 and 5, and both experimental steels were fractured with tough nest fractures.

The microstructure photographs of experimental steels A and B at different tensile temperatures were shown in Figures 6 and 7. When the tensile temperature was 800°C, a large number of small-sized equiaxed grains appear around the fracture of the experimental steel A without Nb, V microalloying, which indicates that dynamic recrystallization of experimental steel A occurs, as shown in Figure 6(a). As the stretching temperature increases, the number of large-sized grains deformed by stretching of experimental steel A gradually decreases and the recrystallization area gradually increases, as shown in Figure 6.

The grains in the experimental steel B with Nb, V microalloying were elongated When the deformation temperature was 800°C, and no dynamic recrystallization was observed, as shown in Figure 7(a). When the deformation temperature was 850°C to 950°C, the experimental steel B was dominated by large-sized grains of tensile deformation, with a small amount of grain nucleation observed at grain boundaries, and we saw an insignificant

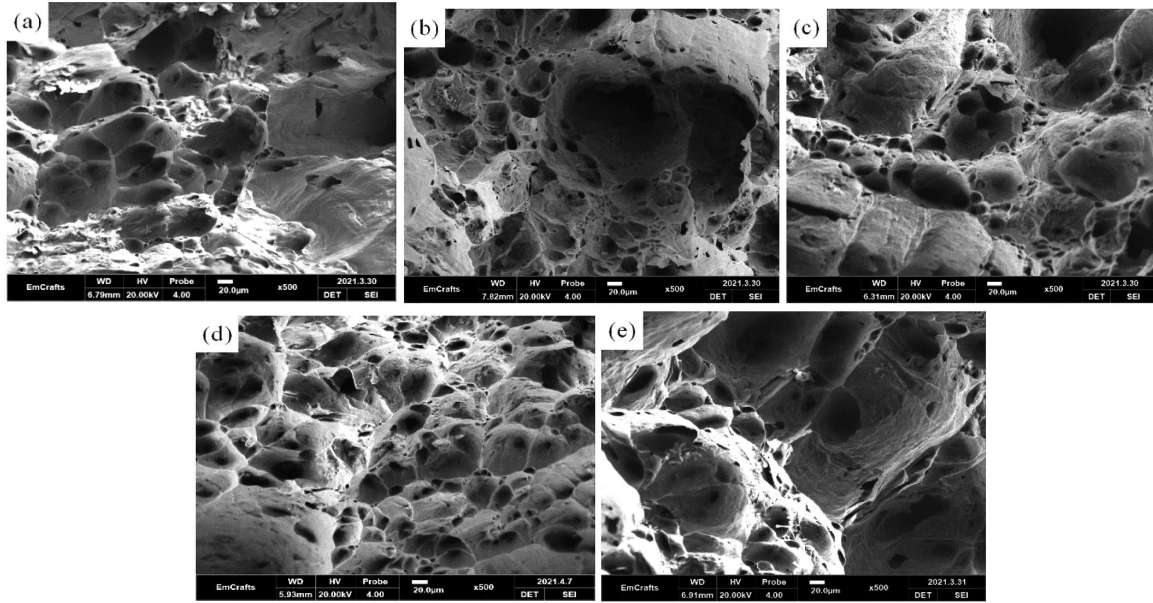


Figure 4: Fracture images of steel A (a)~(e) correspond to 800°C~1000°C (a) 800°C; (b) 850°C; (c) 900°C; (d) 950°C; (e) 1000°C.

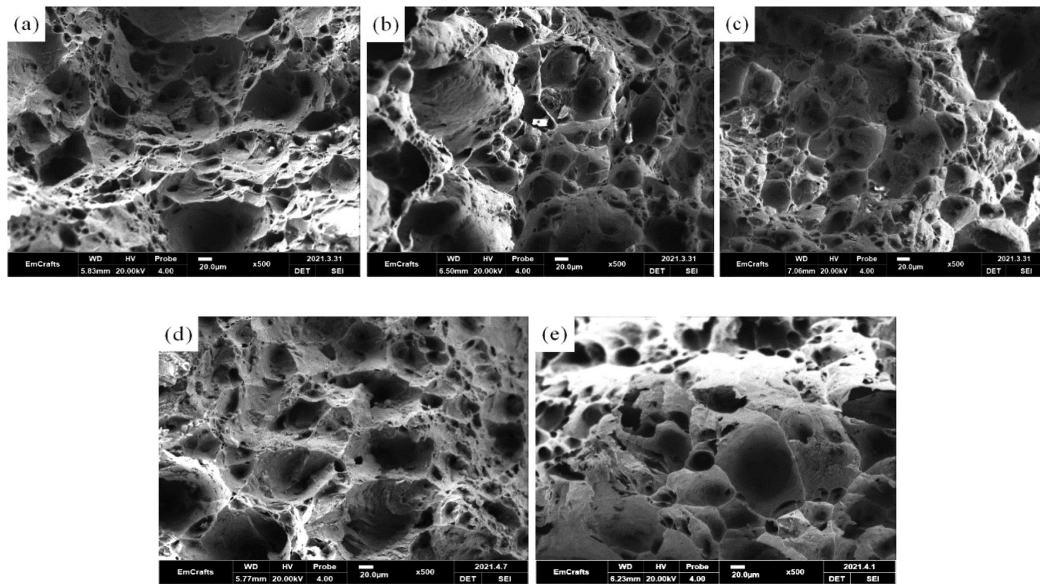


Figure 5: Fracture images of steel B (a)~(e) correspond to 800°C~1000°C (a) 800°C; (b) 850°C; (c) 900°C; (d) 950°C; (e) 1000°C.

nificant degree of recrystallization, as shown in Figures 7(b)~(d). When the deformation temperature exceeded 1000°C, the grains of experimental steel B tensile deformation disappeared, and a large number of small-sized equiaxed grains were observed with a significant degree of recrystallization, as shown in Figures 7(e, f).

The size of recrystallized grains at tensile temperatures above 1000°C was counted, and the size of recrystallized grains of experimental steels A and B at tensile temperatures of 1000°C were 16.62 μm and 13.81 μm, respectively. At the tensile temperature of 1100°C, the recrystallized grain size of experimental steels A and B increased to 30.87 μm and 22.64 μm, and the statistical results showed that the recrystallized grain size of unmicroalloyed experimental steel A was larger than that of microalloyed experimental steel B.

SEM micrographs of the microstructure at the fracture of experimental steels A and B at different deformation temperatures were shown in Figure 8. When the deformation temperature was 900°C, microcracks were observed at the grain boundaries of the fracture tissue of both experimental steels A and B, as shown in Figures 8(a, c). When the deformation temperature was 1100°C, microcracks were observed to be concentrated

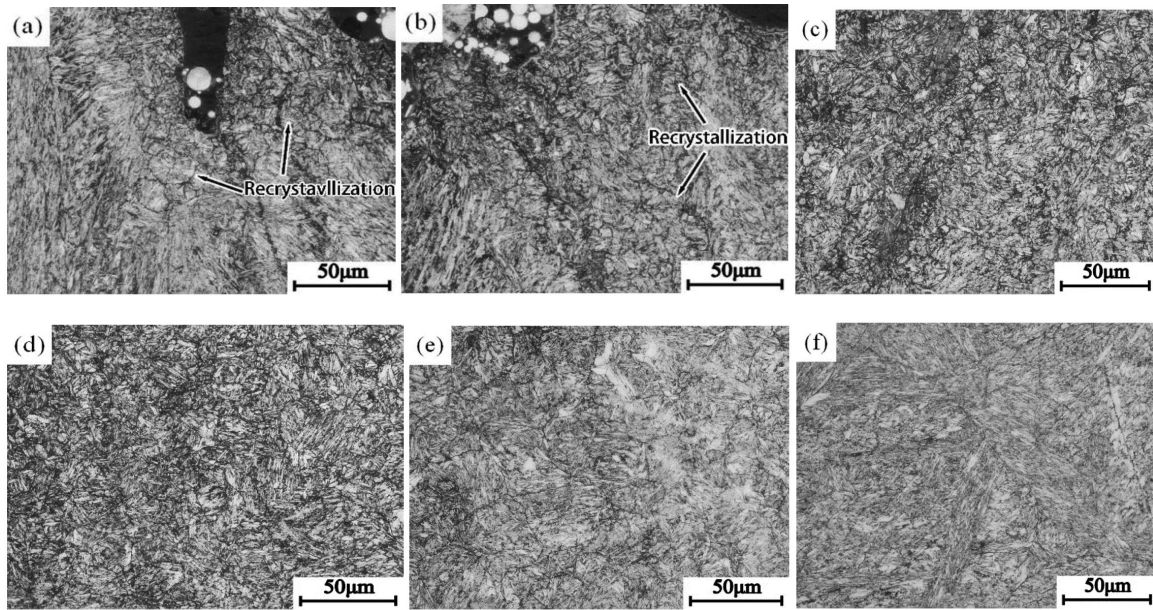


Figure 6: Recrystallization images of steel A (a)~(f) correspond to 800°C~1100°C (a) 800°C; (b) 850°C; (c) 900°C; (d) 950°C; (e) 1000°C; (f) 1100°C.

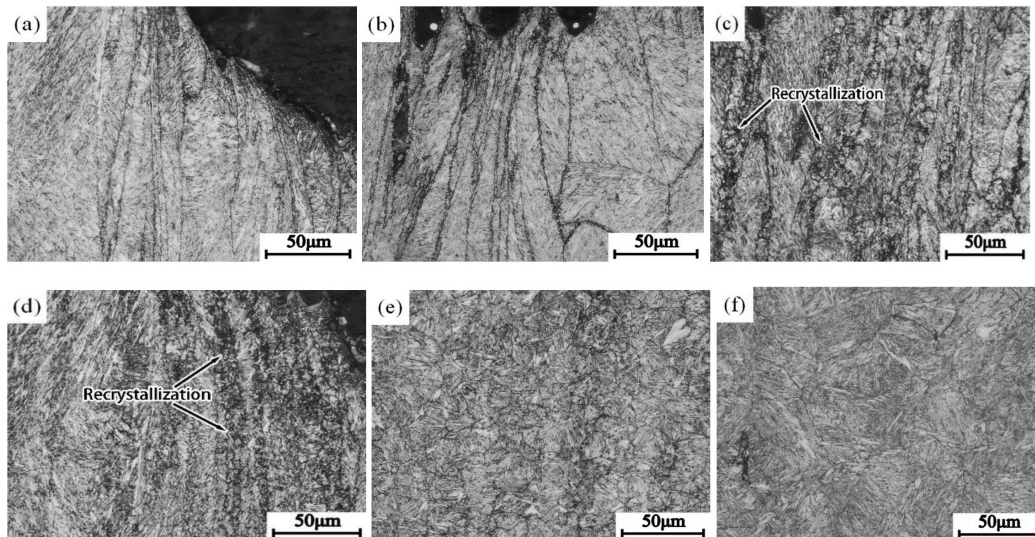


Figure 7: Recrystallization images of steel B (a)~(f) correspond to 800°C~1100°C (a) 800°C; (b) 850°C; (c) 900°C; (d) 950°C; (e) 1000°C; (f) 1100°C.

within the grain for both experimental steels A and B. No microcracks were found at the grain boundaries, as shown in Figures 8(b, d).

3.3. Precipitate phase

The experimental steel B precipitation phase was analyzed by SEM and EDS, as shown in Figure 9. When the tensile temperature of experimental steel B was 900°C, a large-size NbC and a large amount of small-size precipitates were observed, as shown in Figure 9(a). When the tensile temperature was 1100°C, the number of small-size precipitates were significantly reduced, as shown in Figure 9(b). The precipitates of both sizes were analyzed by EDS energy spectroscopy, and the results of EDS analysis were shown in Figures 9(c, d). The results showed that the Nb content was higher and the V content was lower in the two precipitated phases. Previous research shows [7, 8] that the addition of Nb elements to N-free steels tends to produce tens to hundreds of nanometers of NbC precipitation phases, so the two Nb-containing phases are correspondingly NbC precipitation phases. The large size NbC was observed and cracks were found near the large size NbC, as shown in Figures 9(e, f).

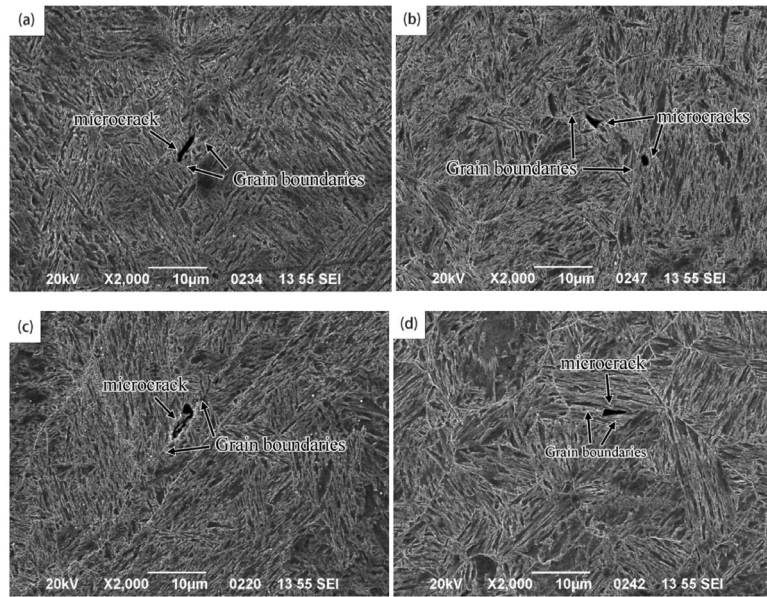


Figure 8: Structures near the fracture of steel at different deformation temperatures (a) steel A at 900°C; (b) steel A at 1100°C; (c) steel B at 900°C; (d) steel B at 1100°C.

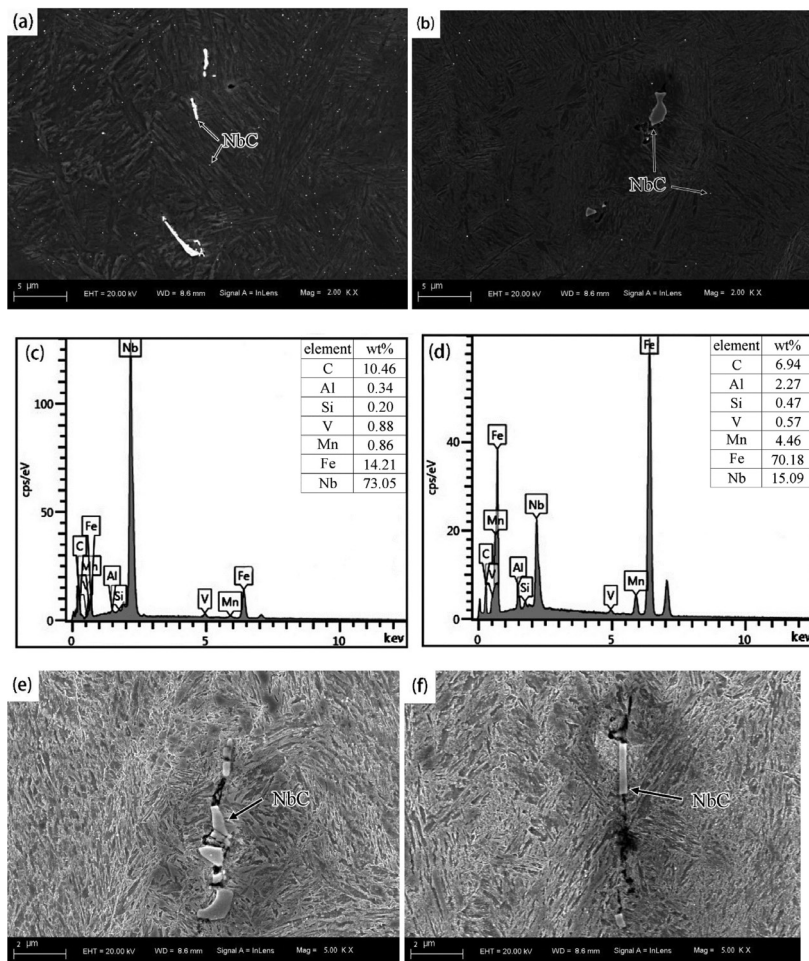


Figure 9: SEM photos and EDS results of precipitates at 900°C and 1100°C (a) 900°C; (b) 1100°C; (c) large-sized precipitated phase; (d) small-sized precipitated phase; (e) 900°C large-sized NbC; (f) 1100°C large-sized NbC.

4. DISCUSSION

As mentioned above, as the tensile temperature increase, the dynamic recrystallization of experimental steels A and B gradually increase, and plasticity is subsequently enhanced. Nb, V microalloying can improve the high-temperature plasticity of the microalloyed steel B above 1000°C while reducing the plasticity of the microalloyed steel B below 1000°C. The small size of NbC precipitated phase can inhibit the dynamic recrystallization of the microalloyed steel B, while the large size of NbC precipitated phase can induce cracks.

Dynamic recrystallization of experimental steels A and B can improve thermoplasticity at high temperatures. As shown in Figure 3(b), significant plasticity gains were observed in the new martensitic low-density steels during the high temperature stretching at 800–1100°C. The degree of dynamic recrystallization of the un-Nb, V microalloyed steel A increases with temperature, allowing the deformation to be dispersed within the grain. This leads to a reduction in the difference in strain within each grain and near the grain boundaries. This reduces the stress concentration and the deformation resistance generated during the deformation process. So the steel can withstand greater deformation before fracture, which increases the plasticity of the steel [12]. On the other hand, as the stretching temperature increases, the cracks shift from the grain boundaries to the inner grain, as shown in Figure 8. Previous research also shows that dynamic recrystallization always occurs preferentially at grain boundaries, which can substantially reduce the stress concentration at grain boundaries and thus inhibit crack formation at grain boundaries [14, 15]. The experimental steel recrystallizes sufficiently at high temperatures, so the rate of grain boundary migration is higher than the rate of microcrack expansion. Therefore, the grains can wrap around the crack and prevent its expansion, which enhances the plasticity of the test steel.

Nb, V microalloying can improve the high-temperature plasticity of the microalloyed steel B above 1000°C, reduce the plasticity of the microalloyed steel B at 1000°C and below. When the deformation temperature is below 1000°C, the microalloyed steel B does not undergo dynamic recrystallization, which resulted in a decrease in its plasticity. When the tensile temperature exceeds 1000°C, dynamic recrystallization has occurred in the microalloyed steel B. Since Nb and V microalloy elements can refine the recrystallized grains[9], the microalloyed steel B plasticity can further improve.

On the other hand, a large amount of small-sized NbC can inhibit the occurrence of dynamic recrystallization in microalloyed steel B. Dynamic recrystallization can occur in un-Nb, V microalloyed steel A at all tensile temperatures, while the significant dynamic recrystallization only occurs in Nb, V microalloyed steel B exceeds 1000°C, as shown in Figure 7. Previous research had shown that the Nb precipitation phase inhibits the occurrence of dynamic recrystallization [13]. According to this theory, when the temperature is below 1000°C, a large number of small-sized NbC precipitated phases exist within microalloyed steel B. The inhibition of dynamic recrystallization by small-sized NbC is greater than the driving force for dynamic recrystallization by high temperature, the dynamic recrystallization does not occur in microalloyed steel B. When the temperature exceeds 1000°C, the number of NbC decreases the inhibition and dynamic recrystallization of microalloyed steel B occurs.

In addition, the large size of NbC promotes cracking, as shown in Figures 9(e, f). When the deformation temperature exceeds 1000°C, the microalloyed steel B recrystallized grains are finer after microalloying, which leads to an increased hindrance to crack expansion. The enhancement of thermoplasticity is stronger than the damage caused by NbC-induced microcracking, therefore the plasticity of the microalloyed steel B is enhanced. When the deformation temperature drops at or below 1000°C, the un-Nb, V microalloyed steel A recrystallizes significantly and the plasticity decreases less. But the microalloyed steel B does not recrystallize significantly, the grain boundary migration rate is lower, which leads to reduced inhibition of microcrack extension, therefore the plasticity of the microalloyed steel B decreases.

5. CONCLUSIONS

Through the study of new martensitic low-density steel thermoplastic, the important results are summarized as follows.

- (1) New martensitic low-density steels have good high-temperature plasticity and no obvious brittle fracture. As the tensile temperature increases, the degree of recrystallization of the experimental steels gradually increases, the effect on crack obstruction gradually enhances, and the plasticity gradually rises.
- (2) Nb, V microalloying can significantly enhance the high-temperature plasticity of the microalloyed steel above 1000°C and reduce the plasticity of the microalloyed steel below 1000°C. When the deformation temperature at 1000°C and below, Nb, V microalloyed steel precipitates a large number of small-sized NbC precipitation phases, which inhibits the occurrence of dynamic recrystallization and leads to a reduction in the inhibition of microcrack extension and a decrease in plasticity. When the deformation temperature exceeds 1000°C, the number of small-sized NbC precipitation phases decreases, the microalloyed steel

recrystallizes sufficiently, and the Nb, V microalloy refines the original and recrystallized grains of the microalloyed steel, which has a greater hindering effect on microcrack expansion and enhanced plasticity.

- (3) Large-size NbC precipitation phase can promote the generation of microcracks, so we should try to avoid the appearance of a large-size NbC precipitation phase.

6. ACKNOWLEDGMENTS

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7. BIBLIOGRAPHY

- [1] DU, Z.L., LIN, B.Q., “Changes in automobile energy consumption during urbanization: evidence from 279 cities in China”, *Energy Policy*, v. 132, pp. 309–317, Sep. 2019. doi: <http://dx.doi.org/10.1016/j.enpol.2019.05.050>
- [2] AGARWAL, J., SAHOO, S., MOHANTY, S., *et al.*, “Progress of novel techniques for lightweight automobile applications through innovative eco-friendly composite materials: a review”, *Journal of Thermoplastic Composite Materials*, v. 33, n. 7, pp. 978–1013, Jul. 2020. doi: <http://dx.doi.org/10.1177/0892705718815530>
- [3] CHEN, M.X., LIU, W.D., LU, D.D., “Challenges and the way forward in China’s new-type urbanization”, *Land Use Policy*, v. 55, pp. 334–339, Sep. 2016. doi: <http://dx.doi.org/10.1016/j.landusepol.2015.07.025>
- [4] ZAMBRANO, O.A., “A general perspective of Fe-Mn-Al-C steels”, *Journal of Materials Science*, v. 53, n. 20, pp. 14003–14062, Oct. 2018. doi: <http://dx.doi.org/10.1007/s10853-018-2551-6>
- [5] PALMIERE, E.J., GARCIA, C.I., DEARDO, A.J., “Compositional and microstructural changes which attend reheating and grain coarsening in steels containing Niobium”, *Metallurgical and Materials Transactions A*, v. 25, n. 2, pp. 277–286, Feb. 1994. doi: <http://dx.doi.org/10.1007/BF02647973>
- [6] YANG, C., “Recent development and applications of vanadium microalloying technology”, *Journal of Iron and Steel Research*, v. 32, n. 12, pp. 1029–1043, Feb. 2020. doi: <https://doi.org/10.13228/j.boyuan.issn1001-0963.20200200>
- [7] SHANMUGAM, S., MISRA, R.D.K., MANNERING, T., *et al.*, “Impact toughness and microstructure relationship in niobium- and vanadium-microalloyed steels processed with varied cooling rates to similar yield strength”, *Materials Science and Engineering: A*, v. 437, n. 2, pp. 436–445, Nov. 2006. doi: <http://dx.doi.org/10.1016/j.msea.2006.08.007>
- [8] ZHOU, P., DU, L., ZHOU, M., *et al.*, “Microstructure characteristics and mechanical properties of Nb-Mo and Nb micro-alloying hot-rolled X100 pipeline steels”, *Iron and Steel*, v. 47, n. 9, pp. 63–67, Feb. 2012. doi: <https://doi.org/10.13228/j.boyuan.issn0449-749x.2012.09.006>
- [9] FU, L.M., SHAN, A.D., WANG, W., “Effect of Nb solute drag and NbC precipitate pinning on the recrystallization grain growth in low carbon Nb-microalloyed steel”, *Chin Shu Hsueh Pao*, v. 46, n. 7, pp. 832–837, Jul. 2010. doi: <http://dx.doi.org/10.3724/SP.J.1037.2010.00832>
- [10] GURGEL, M.A.M., BAÊTA JÚNIOR, E.S., TEIXEIRA, R.S., *et al.*, “Caracterização microestrutural de uma liga Fe-7,1Al-0,7Mn-0,4C-0,3Nb do sistema Fe-Mn-Al-C”, *Matéria (Rio de Janeiro)*, v. 27, n. 2, pp. e2021464000, Apr. 2022. doi: <http://dx.doi.org/10.1590/1517-7076-rmat-2021-46400>
- [11] DU, H., CAI, J.H., WANG, Y.S., *et al.*, “Effect of partial recrystallization on microstructure and tensile properties of NiFeCoCrMn high-entropy alloy”, *Transactions of Nonferrous Metals Society of China*, v. 32, n. 3, pp. 947–956, Mar. 2022. doi: [http://dx.doi.org/10.1016/S1003-6326\(22\)65841-2](http://dx.doi.org/10.1016/S1003-6326(22)65841-2)
- [12] CUI, H., CHEN, D.Y., ZHANG, K.T., “Effects of Al content on the high-temperature mechanical properties of Al-TRIP steel”, *Materials Science and Technology*, v. 36, n. 4, pp. 484–491, Mar. 2020. doi: <http://dx.doi.org/10.1080/02670836.2019.1710928>
- [13] LIU, J., CHENG, Z., HUANG, Y., *et al.*, “High temperature thermoplasticity of niobium microalloyed 1800 MPa grade hot forming steels”, *Iron & Steel*, v. 23, pp. 1–13, Apr. 2023. DOI: <https://doi.org/10.13228/j.boyuan.issn0449-749x.20230056>
- [14] DOHERTY, R.D., HUGHES, D.A., HUMPHREYS, F.J., *et al.*, “Current issues in recrystallization: a review”, *Materials Science and Engineering: A*, v. 238, n. 2, pp. 219–274, Nov. 1997.
- [15] WANG, Z.H., SUN, S.H., WANG, B., *et al.*, “Effect of grain size on dynamic recrystallization and hot-ductility behaviors in high-Nitrogen CrMn austenitic stainless steel”, *Metallurgical and Materials Transactions A*, v. 45, pp. 3631–3639, Jul. 2014. doi: <http://dx.doi.org/10.1007/s11661-014-2290-5>