High-temperature hardness in steels with various carbon concentrations and microstructures measured by small ball rebound hardness test

Norimitsu Koga[1], Kouki Koizumi[2], Shuto Takayasu[2], Osamu Umezawa[3,4], Mizuki Watanabe[5], Masayuki Yamamoto[5], Takashi Yamamoto[5]

1 Faculty of Mechanical Engineering, Institute of Science and Engineering, Kanazawa University, Kakumamachi, Kanazawa, Ishikawa, 920-1192, Japan.
2 Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, 240-8501, Japan.
3 Faculty of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, 240-8501, Japan.
4 Center of Advanced Innovation Technologies, Vysoká Škola Báňská - Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic.
5 Yamamoto Scientific Tool Laboratory Co., Ltd. 2-15-4 Sakae-cho Funabashi Chiba, 273-0018 Japan.

Keywords: *high temperature hardness test, interstitial free steel, ferritic steel, pearlitic steel, neutron diffraction.*

1. Introduction

Recently, a small ball rebound hardness test was developed as a handheld and convenient device for rebound hardness test. This method measures the hardness from the ratio of the velocities of the impact ball after and before the impact on the specimen surface, and the measurement time is so short that heat cannot be transferred from the specimen surface into the impact ball. Thus, it can be applied to any temperatures from cryogenic to high temperatures without specific equipment. In this study, high-temperature hardness in steels with various carbon concentrations and microstructures was measured using small ball rebound hardness test. Based on the obtained results, the origin of the characteristic hardening at high temperature and the relationship between microstructure and high-temperature hardness were discussed.

2. Experimental procedure

2.1 Materials

Four types of steels were used in this experiment: IF steel (Fe-0.002%C-0.046%Ti, in mass%), ULC steel (Fe-0.011%C), SK85 steel (Fe-0.85%C-0.23%Si-0.35%Mn), and hypereutectoid steel (Fe-1.36%C-0.20%Si-0.24%Mn). The ULC steel was heat-treated at 1273 K for 0.6 ks and subsequently furnace-cooled to precipitate the cementite completely. The SK 85 steel was heat-treated at 1123 K for 0.6 ks and subsequently air cooled to precipitate the pearlite (pearlitic steel). The hypereutectoid steel was solution treated at 1273 K for 1.8 ks and air cooling for making full pearlite structure (hypereutectoid pearlite steel). The hyperutectoid steel with full pearlite structure was heat-treatment at 1073 K for 86.4 ks and furnace cooling for spheroidizing the cementite (spheroidized cementite steel).

2.2 Small ball rebound hardness test

The small ball rebound hardness test (eNM3A10, Yamamoto Scientific Tool Laboratory Co., Ltd) was conducted five times at each temperature level from 173 K to 1273 K, and the specimen was reheated at the test temperature for 300 s in each test interval.

2.3 *In-situ* neutron diffraction measurement during heating.

The in-situ neutron diffraction measurements during heating from 323 K to 1173 K were carried out using "TAKUMI", a time-of-flight engineering materials diffractometer at the Japan-Proton Accelerator Research Complex (J-PARC). The heating rate was 0.05 K/s and the temperature was held constant for 0.3 ks at every 100 K from 473 K to 1073 K. The holding time for pearlitic steel was 1.8 ks. The diffractometer operated in the time-of-flight diffraction mode, using neutron pulses with a range of energies. The lattice parameters were obtained using the Pawley refinement that utilized the Z-Rietveld software.

3. Results and discussion

3.1 Characteristic hardening at high temperature

Figure 1 shows the coefficient of restitution corresponding to the hardness in small ball rebound hardness test as a function of temperatures in IF, ULC and pearlitic steels[1)]. Until 700 K, the coefficient of restitution continuously decreased with increasing temperature in all steels, which is coincident with general temperature dependence of hardness in metal materials. However, within the range from 700 K to 1000 K, characteristic hardening occurred in ULC and pearlitic steels: the coefficient of restitution increased with increasing temperature. On the other hand, IF steel with no solute carbon exhibited a continuous decrease of the coefficient of restitution even above 700 K. Figure 2 shows the difference in lattice parameter in ULC and pearlitic steels from that of IF steel measured by *In-situ* neutron diffraction during raising temperature[1)]. The lattice parameter in ULC steel became high above 700 K compared to that in IF steel. Furthermore, the difference in lattice parameter in pearlitic steel was decreased until 700 K and then leveled off, which also indicates the increase of lattice parameter. Therefore, it can be concluded that the increase of lattice parameter above 700 K in ULC and pearlitic steels is due to the solid solute of ultra-low carbon (approximately 100 ppm) and that provided the characteristic hardening.

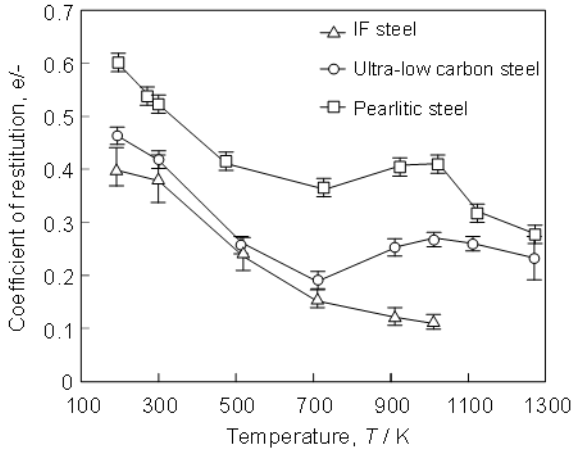

Figure 1 Coefficient of restitution as a function of temperature in IF, ultra-low carbon and pearlitic steels[1].

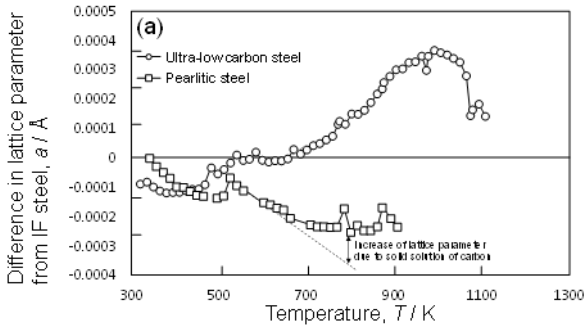

Figure 2 (a) Difference in the lattice parameter from IF steel as a function of temperature[1].

3.2 Effect of microstructure on high-temperature hardness.

Figure 3 shows the coefficient of restitution in as-received hypereutectoid, hypereutectoid pearlite, and spheroidized cementite steels. Hypereutectoid pearlitic steel exhibited the highest coefficient of restitution among hypereutectoid steels[2]. As-received hypereutectoid steel consisted of ferrite, coarse spheroidized cementite and a small amount of graphite had higher hardness than the spheroidized cementite steels consisted of ferrite, fine spheroidized cementite and a large amount of graphite. Table 1 shows the volume fraction of each phase calculated from the area fraction of graphite measured from optical microscopy images[2]. The increase of volume fraction of graphite significantly decreased the volume fraction of cementite phase. Table 2 summarizes the estimated hardness at 673 K from following equation (1) based on the rule of mixture and measured hardness converted from the coefficient of restitution to Vickers hardness[2].

$$HV_{all} = HV_a V_\alpha + HV_{Fe_3C} V_{Fe_3C} + HV_C V_C \quad (1)$$

α, Fe_3C, and C denote ferrite, cementite, and graphite, respectively. Vickers hardness of the ferrite phase at 673 K adopted the measured hardness of the ULC steel (40 HV), and that of the cementite phase is 520 HV according to Umemoto's work[3]. The Vickers hardness of graphite at 673 K could not be found and 50 HV, which is the hardness at room temperature, was adopted. The estimated Vickers hardness was almost the same as the measured hardness in the as-received and spheroidized cementite steels. It suggests that the rule of mixture was satisfied in these steels. Furthermore, it can be concluded that the lowest hardness in the spheroidized cementite steel was due to the large volume fraction of graphite. The measured hardness in the pearlite steel was much higher than the estimated hardness, suggesting that the pearlite structure does not obey the rule of the mixture.

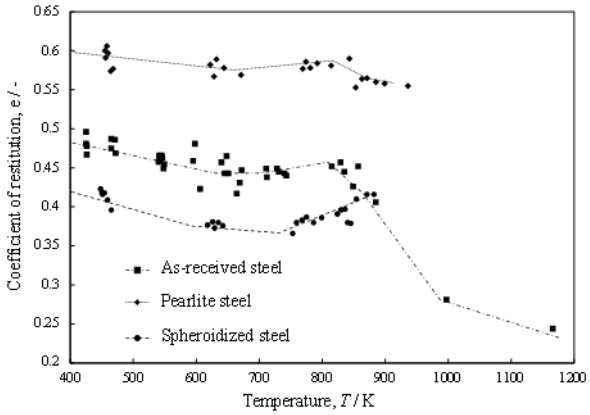

Figure 3 Coefficient of restitution as a function of temperature in as-received, pearlite and spheroidized cementite steels[2].

Table 1 Volume fraction of each phase estimated from area fraction of graphite (Table 1) and equilibrium diagram of Fe-C binary alloy[2].

	As-received	Pearlite	Spheroidized cementite
Ferrite (%)	81.3	80.5	86.5
Cementite (%)	18.5	19.5	11.5
Graphite (%)	0.2	0.0	2.0

Table 2 Vickers hardness (*HV*) at 673 K converted from coefficient of restitution and estimated Vickers hardness (HV_{all}) from the role of mixture[2].

	As-received	Pearlite	Spheroidized cementite
HV at 673 K converted from coefficient of restitution	131	216	95
Estimated HV_{all}	130	134	95

4. Conclusion

The characteristic hardening occurred above 700 K in the carbon steels, which is due to the solid solution of ultra-low carbon. The estimated hardnesses from the hardness and volume fraction of each phase was almost same with measured hardnesses in as-received hypereutectoid and spheroidized cementite steels. The presence of graphite remarkably decreased the high-temperature hardness. Therefore, the state of carbon is important for the high-temperature hardness.

Acknowledgments

The neutron diffraction experiments were performed using BL19 in the Materials and Life Science Experimental Facility (MLF) of J-PARC following the proposal of 2018P0213.

Part of this work was financially supported by Iketani Science and Technology Foundation (0311099-A), the Grant-in-Aid for Scientific Research (KAKENHI) Grant No. 20K14605, and JKA and its promotion funds from KEIRIN RACE

References

1) N.Koga, O. Umezawa, M. Yamamoto, T. Yamashita, S. Morroka, T. Kawasaki, S. Harjo: Met. Mater. Trans. A, 52(2021) 897-901.
2) N.Koga, K. Koizumi, S. Takayasu, O. Umezawa, M. Watanabe, M. Yamamoto, T. Yamamoto: Mater. Trans., 64(2023) 1600-1604.
3) M. Umemoto, Z.G. Liu, K. Masuyama and K. Tsuchiya: Scr. Mater., 45(2001), p. 391.